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ENGINEERING PROPERTY DATA

ON ROCKET PROPELLANTS

Annual Summary Report

By

Chemical and Material Sciences Department
Research Division of Rocketdyne
A Division of North American Aviation, Inc.
6633 Canoga Avenue, Canoga Park, California

TECHNICAL REPORT AFRPL TR-67-151

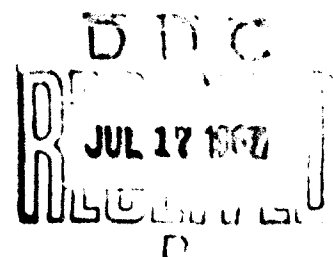
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**Air Force Rocket Propulsion Laboratory
Research and Technology Division
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FOREWORD

- (U) This is an interim final progress report submitted under G.O. 07119 in compliance with Contract AF04(611)-11407, Part I.D.1 and Line items 6 and 7 of DDI423. The research reported herein, which covers the first 12 months (1 April 1966 through 31 March 1967) of a 24-month program, was sponsored by the Air Force Rocket Propulsion Laboratory, Research and Technology Division, Air Force Systems Command, Edwards, California, with Mr. G. Allen Beale acting as the Air Force Project Engineer.
- (U) This program was conducted by the Propellant Technology function of the Rocketdyne Research Division, with Dr. E. F. C. Cain serving as Program Manager and Mr. M. T. Constantine serving as the Responsible Project Scientist.
- (U) This report has been assigned the Rocketdyne identification number R-7029.
- (U) The following technical personnel contributed to the work described in this report:

Phase I: Literature Search

K. J. Youel

Phase II: Experimental Determinations

Dr. A. Axworthy (Specific Heat)

Dr. J. Gerhauser (Specific Heat)

Dr. J. V. Hamilton (Specific Heat)

J. V. Lecce (Thermal Conductivity, Viscosity)

R. W. Melvold (Specific Heat)

J. Quaglino (Phase Properties, Sonic Velocity)

Dr. S. E. Rodriguez (Viscosity, Inert Gas Solubility)

M. J. Seric (Viscosity, Inert Gas Solubility)

Dr. W. Unterberg (Phase Properties, Sonic Velocity,
Inert Gas Solubility, Thermal Conductivity,
Viscosity)

Phase III: Evaluation and Compilation of Data

M. M. Williams

K. J. Youel

This technical report has been reviewed and is approved.

W. H. EDELKE, Colonel, USAF
Chief, Propellant Division

ABSTRACT

- (U) The results of the initial 12-month period of the current 24-month program on the analytical and experimental characterization of the physical properties of selected liquid propellants are presented in three phases. In Phase I, a continuous review of the literature was conducted to ensure the acquisition and documentation of the latest possible propellant properties data for evaluation and possible inclusion into a propellant properties handbook. Phase II experimental efforts have resulted in the measurement of chlorine trifluoride density and vapor pressure; sonic velocity in chlorine trifluoride and chlorine pentafluoride; nitrogen gas solubility in chlorine pentafluoride; specific heat of UDMH, 50 w/o hydrazine-50 w/o UDMH, and MHF-3; thermal conductivity of UDMH, MHF-3, and MHF-5; and viscosity of chlorine pentafluoride. Phase III efforts included the evaluation and assembly of all data generated in Phases I and II, preparation of complete physical property bibliographies for diborane and hydrazine, and assembly of hydrazine physical properties data.

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INTRODUCTION

- (U) Under Contract AF04(611)-10546, Rocketdyne initiated a 12-month analytical and experimental program on the rational and systematic physical characterization of selected liquid rocket propellants. This program was designed as a primary step in the elimination of the data gaps which have hindered propellant utilization and development in the past. The overall program objective was to assemble and experimentally complete the data on essential physical properties of current and near-term liquid rocket propellants over temperature and pressure ranges of practical use to propulsion engineering.
- (U) This initial program, conducted in three interrelated phases, was completed on 31 March 1966 (Ref. 1). During the effort, a compilation of physical properties data was made as a result of an extensive literature survey. Experimental efforts resulted in the measurement of: (1) the thermal conductivity of $N_2H_4-(CH_3)_2N_2H_2$ (50-50) and $CH_3N_2H_3$; (2) IRFNA and ClF_5 sonic velocity (and calculation of compressibility); (3) ClF_3 and $CH_3N_2H_3$ specific heat, and correction of ClF_5 specific heat data; (4) ClF_3 phase properties; and (4) the design and assembly of apparatus for measurement of inert gas solubility in liquids and liquid viscosities at extended temperatures and pressures. Analytical efforts included the assembly and evaluation of physical property data on MHP-1, MHP-3, MHP-5, ClF_3 , and ClF_5 for future correlation and summary publication.
- (U) The present three-phase program, being conducted under Contract AF04(611)-11407, represents a 24-month extension and expansion of the objectives of the previous effort. Phase I effort consists of a continuous review of the current literature to document reported propellant properties. In Phase II, effort is directed at the experimental determination of unavailable engineering data for selected oxidizers and fuels which are required to document reported propellant properties. Effort under Phase III includes the compilation, correlation, and evaluation of all data obtained from

Phases I and II and presentation of the valid data in an annual technical report.

- (U) This report describes each phase of the present program in terms of the objective and summarizes the results and accomplishments achieved during the first 12 months.

SUMMARY

- (U) Analytical and experimental research conducted during the initial 12-month period of the current 24-month program on the rational and systematic physical characterization of selected liquid rocket propellants is described in three phases.
- (U) Phase I consisted of a continuous review of the current literature and efforts of other investigators in the field to ensure the acquisition and documentation of the latest possible propellant properties data for evaluation and possible inclusion into a propellant properties handbook. During this survey, a preliminary screening of 3894 reports resulted in the detailed review of 461 reports for potentially pertinent data.
- (U) The experimental characterization of essential physical properties of selected propellants was conducted under Phase II. Experimental efforts were directed at measurements of phase properties, sonic velocity (and compressibility), inert gas solubility, specific heat, thermal conductivity, and viscosity of selected propellants in an order related to their importance to the Air Force. During the initial 12-month period of the present contract, Phase II experimental efforts have resulted in the measurement of chlorine trifluoride density and vapor pressure; sonic velocity in chlorine trifluoride and chlorine pentafluoride; nitrogen gas solubility in chlorine pentafluoride; specific heat of UDMH, hydrazine-UDMH (50-50) and MHP-3; thermal conductivity of UDMH, MHP-3 and MHP-5; and viscosity of chlorine pentafluoride.
- (U) A Poole-Nyberg densimeter was used to extend the saturated liquid ClF_3 density data to the temperature range of -22 to -8 C (-8 to 18 F). These data were correlated with previous data and curve fit over a temperature range of -22 to 161 C (-8 to 322 F) with the following equations:

$$\rho(\text{gm/cc}) = 4.924 - 2.517 \times 10^{-2} T(\text{K}) + 7.38429 \times 10^{-5} T(\text{K})^2 - 8.213 \times 10^{-8} T(\text{K})^3$$

and

$$\rho(\text{lb/cu ft}) = 121.360 - 1.226 \times 10^{-1} t_{(F)} + 2.127 \times 10^{-4} t_{(F)}^2 - 8.850 \times 10^{-7} t_{(F)}^3$$

- (U) Vapor pressure data for ClF_3 were obtained over the temperature range of 42.9 to 147.6 C (108 to 298 F), using a constant-volume vapor pressure bomb. Additional vapor pressure data are being determined in this and the critical temperature region to extend presently available data.

- (U) Measurements of sonic velocity in liquid ClF_5 and ClF_3 were conducted at saturated liquid conditions and under pressures of 500 and 1000 psia. The data for saturated liquids were curve fit with the following equations over the indicated temperature ranges:

$$\underline{\text{ClF}_5} \quad c(\text{m/sec}) = 1755 - 4.074 T_{(K)} + 5.936 \times 10^{-4} T_{(K)}^2 \quad (-77 \text{ to } 69 \text{ C})$$

and

$$c(\text{ft/sec}) = 5758 - 7.426 T_{(R)} + 6.011 \times 10^{-4} T_{(R)}^2 \quad (-107 \text{ to } 156 \text{ F})$$

$$\underline{\text{ClF}_3} \quad c(\text{m/sec}) = 1951.8 - 3.7508 T_{(K)} \quad (-59.7 \text{ to } 74.5 \text{ C})$$

and

$$c(\text{ft/sec}) = 6401.8 - 6.8348 T_{(R)} \quad (-75.5 \text{ to } 166.1 \text{ F})$$

- (U) Adiabatic compressibilities calculated from these data established the following relationships for saturated liquid ClF_5 and ClF_3 :

$$\underline{\text{ClF}_5} \quad \beta_{(\text{atm}^{-1})} = 1.1563 \times 10^{-4} + 1.3942 \times 10^{-6} t_{(C)} + 1.2708 \times 10^{-8} t_{(C)}^2 + 1.4680 \times 10^{-10} t_{(C)}^3 + 9.6853 \times 10^{-13} t_{(C)}^4$$

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and

$$\beta_{(\text{psia}^{-1})} = 6.4065 \times 10^{-6} + 4.0065 \times 10^{-8} t_{(F)} + \\ 1.4103 \times 10^{-10} t_{(F)}^2 + 9.0915 \times 10^{-13} t_{(F)}^3 + \\ 6.2782 \times 10^{-15} t_{(F)}^4$$

$$\text{ClF}_3 \quad \beta_{(\text{atm}^{-1})} = 6.2594 \times 10^{-3} + 6.1059 \times 10^{-5} t_{(C)} + \\ 3.7345 \times 10^{-7} t_{(C)}^2 + 2.6649 \times 10^{-9} t_{(C)}^3 + \\ 2.1283 \times 10^{-11} t_{(C)}^4$$

and

$$\beta_{(\text{psia}^{-1})} = 3.5921 \times 10^{-4} + 1.8837 \times 10^{-6} t_{(F)} + \\ 5.7058 \times 10^{-9} t_{(F)}^2 + 1.3434 \times 10^{-11} t_{(F)}^3 + \\ 1.3795 \times 10^{-13} t_{(F)}^4$$

- (U) As a result of measurements of nitrogen gas solubility in ClF_5 , a 90 F solubility point of 2.35×10^{-5} lb N_2 /lb ClF_5 -psi was established at a total pressure of 350 psia. A set of data at 120 F contained values from 2.58×10^{-5} to 3.28×10^{-5} lb N_2 /lb ClF_5 -psi for total pressures from 400 to 910 psia.
- (U) Specific heat measurements were made on UDMH, N_2H_4 -UDMH(50-50), and MHP-3 using an adiabatic calorimeter. The specific heat of UDMH was determined over a temperature range of 1.7 to 70.5 C (35 to 159 F); however, some discrepancies were noted in the data with the use of different apparatus sample chambers. The specific heat data resulting from measurements on N_2H_4 -UDMH(50-50) were curve fit over the temperature range 2 to 38 C (36 to 100 F) to the following equation:

$$C_p(\text{cal/g-C}) = 0.715 + 0.00047 t_{(C)}$$

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Preliminary measurements of MHF-3 specific heat were conducted from -47.8 to 24.9 C (-54 to 77 F). A determination of N_2H_4 specific heat, used for calibration purposes, agreed with previously established data.

- (C) Thermal conductivity measurements were conducted on UDMH, MHF-3, and MHF-5 fuels through the use of a steady-state concentric-cylinder conductivity cell. The valid data for UDMH were curve fit from 0.5 to 251 F with the equation:

$$k(\text{Btu/hr-ft-F}) = 0.1014 - 1.368 \times 10^{-4} t(F)$$

The thermal conductivity data for MHF-3 over the temperature range 0.4 to 251.3 are represented by the equation:

$$k(\text{Btu/hr-ft-F}) = 0.1688 - 1.063 \times 10^{-4} t(F) - 1.891 \times 10^{-7} t(F)^2$$

Preliminary results for the thermal conductivity of MHF-5 indicate values from 0.188 to 0.171 Btu/hr-ft-F over the temperature range 0.5 to 201 F.

- (U) Final assembly, check-out, and calibration of an all-metal capillary viscometer have been completed. Two viscosity data points have been obtained for saturated liquid ClF_5 at 154 F (0.215 cp) and 176 F (0.185 cp). Additional measurements are being obtained in the present viscometer to provide viscosity data over the complete temperature range.
- (U) Phase III involved the assembly of all data generated in Phases I and II, verification of all the data sources, critical evaluation and comparison of conflicting data, and tabulation and correlation of the results. As a result of these efforts, complete physical property bibliographies were compiled for B_2H_6 and N_2H_4 . In addition, effort was initiated on compilation of a bibliography of N_2O_4 physical properties. The N_2H_4 physical property bibliography was used to prepare a set of physical property data sheets on N_2H_4 .

TECHNICAL PROGRAM

PHASE I: LITERATURE SEARCH

OBJECTIVE

- (U) The Phase I objective is the maintenance of a continuous review of the current literature and efforts of other investigators in the field to ensure the acquisition and documentation of the latest possible propellant properties data for evaluation and possible inclusion into a propellant properties handbook. This survey is designed to include, but not necessarily be limited to, the properties of the following fuels and oxidizers:

Oxidizers

Liquid Oxygen (LO_2)
Chlorine Pentafluoride (ClF_5)
Chlorine Trifluoride (ClF_3)
Fluorine (F_2)
Hydrogen Peroxide (H_2O_2)
Nitrogen Tetroxide (N_2O_4)
Mixed Oxides of Nitrogen
(N_2O_4 -NO)
FLOX Mixtures (O_2 - F_2)
Oxygen Difluoride (OF_2)
Tetrafluorohydrazine (N_2F_4)

Fuels

Liquid Hydrogen (LH_2)
 N_2H_4 -UDMH(50-50)
Hydrazine (N_2H_4)
MMH ($\text{CH}_3\text{N}_2\text{H}_3$)
UDMH [$(\text{CH}_3)_2\text{N}_2\text{H}_2$]
 N_2H_4 -MMH mixtures
Hybaline B-3

Alumizine
Pentaborane (B_5H_9)
Diborane (B_2H_6)
MHF fuels
MAF fuels

RESULTS AND ACCOMPLISHMENTS

- (U) A formal survey of current propellant literature, which was initiated under Contract AF04(611)-10546 (Ref. 1), was continued as Phase I of the present program. This survey, which includes the location, acquisition, and documentation of all available propellant properties data of interest to the Air Force, was originally directed at a comprehensive review of physical properties data. However, under the present contract, the survey has been extended to additional engineering properties data.
- (U) The literature survey is being accomplished through two different techniques. One part of the effort is directed at the survey of all reports acquired by Rocketdyne through normal distribution channels. Each of these reports are surveyed with respect to subject matter, and reports containing potential propellant properties data are selected for detailed review. All pertinent data contained in these reports are documented for assembly under Phase III.
- (U) To ensure a complete awareness of all available propellant properties data and their subsequent acquisition and documentation during the current program, this report survey effort is supplemented by a continuous survey of the current releases of Chemical Abstracts, NASA CSTAR Abstracts, Chemical Propulsion Information Agency (CPIA) Abstracts, the Defense Documentation Center (DDC) Technical Abstract Bulletin (TAB), the NBS Cryogenic Data Center's Current Awareness Service, and propellant manufacturers' bulletins. Any pertinent reports located through these abstract sources that have not been acquired previously by Rocketdyne are ordered immediately and eventually reviewed in detail.
- (U) During the first year of the current program, 3894 reports were surveyed; of this total, 461 reports were reviewed in detail for propellant properties data. Pertinent data contained in these reports are being compiled and evaluated under Phase III.

PHASE II: EXPERIMENTAL DETERMINATIONS

OBJECTIVE

- (U) The objective of Phase II is the experimental characterization of essential physical properties of selected liquid propellants. This phase essentially constitutes a 24-month continuation of the efforts initiated under Phase II of Contract AF04(611)-10546 (Ref. 1). Selection of the propellants and properties to be experimentally characterized is related to the unavailability of required data and relative importance of the data to the Air Force. Initial efforts have emphasized the completion of those propellant properties recommended for initial characterization under the previous program. Additional efforts are continuing in an order related to the importance of the data to the Air Force as determined by the Air Force Project Engineer.
- (U) The selected properties are being determined over the liquidus temperature range and over a pressure range of 14.7 psi to 1000 psi, where practical. Changes to the selected list can be made at any time during the program through mutual agreement of Rocketdyne and the Air Force Contracting Officer. Standard test methods are used if available. Wherever unique or new test methods are used, their use has been approved by the Air Force Contracting Officer.

RESULTS AND ACCOMPLISHMENTS

- (U) During the initial 12 months of the current 24-month program, Phase II efforts were directed at the measurement of phase properties, sonic velocity (and calculation of compressibility), inert gas solubility, specific heat, thermal conductivity, and viscosity of selected propellants. The apparatus and techniques used in these measurements were essentially those used previously in similar efforts conducted under Contract AF04(611)-10546 (Ref. 1); however, some further modifications and development of the inert gas solubility,

specific heat, and viscosity apparatus were necessary to improve the quality and accuracy of the measurements. The efforts and results in each of these areas of study are described in the following paragraphs. Included in the discussions are details of the changes in experimental apparatus or methods instituted during the current program.

Phase Properties

- (U) Phase property measurements have been designed to expand the available experimental data on chlorine trifluoride (ClF_3) density and vapor pressure. Density measurements on saturated liquid ClF_3 over a temperature range of -22°C (-8°F) to -8°C (18°F) extended the low-temperature extremity of the previously available data (Ref. 1 and 2). Additional ClF_3 vapor pressure measurements were conducted over the temperature range of 42.9°C (109°F) to 147.6°C (298°F) to increase the accuracy of previously available (Ref. 1 and 3) vapor pressure data in the high-temperature and critical region.
- (U) The experimental apparatus used in the density measurements has been described previously (Ref. 1). A diagram of the apparatus, which was constructed from the design of Poole and Nyberg (Ref. 4), is presented in Fig. 1. This densimeter operates on the principle that a sudden rise in pressure (sensed by the pressure transducer) will occur when all vapor in the variable volume propellant cell is forced to condense by mechanical reduction of the cavity volume containing both liquid and vapor. The volume of the cell (and thus the contained liquid) at this point is indicated by the position of the micrometer attached to the bellows and a prior calibration of the micrometer (defining the micrometer setting-cell volume relationship) of the apparatus with liquid of known density. The density of the sample at this point is then calculated from the weight of the propellant sample and the indicated volume.
- (U) The apparatus is constructed entirely of 300 series stainless steel and is capable of withstanding pressures up to 1500 psi. The volume range

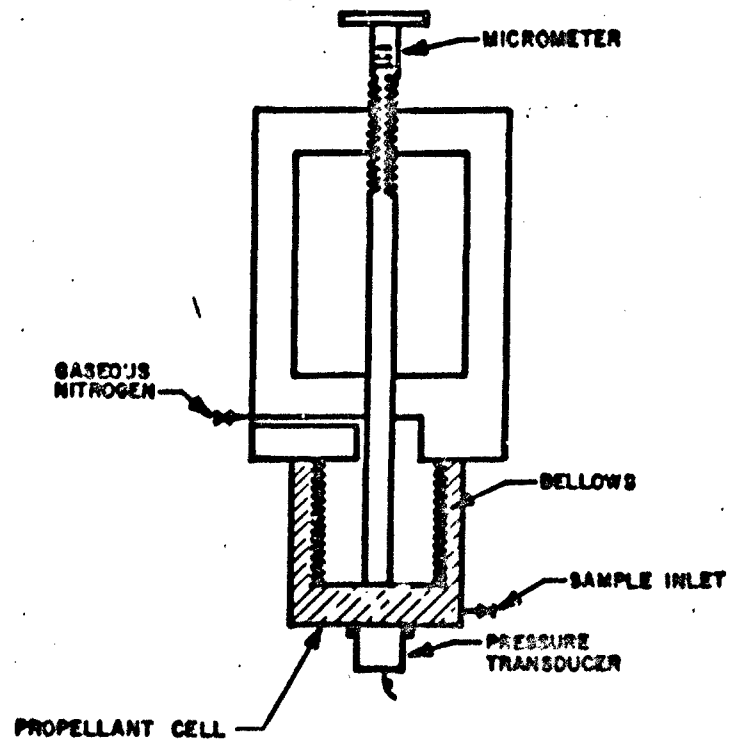


Figure 1. Poole-Nyberg Densimeter.

provided by the stainless-steel bellows permits density measurements over a wide range of temperatures without change of the propellant sample size. The densimeter is placed in a constant-temperature bath for temperature selection and regulation. The temperature of the propellant sample is indicated by a chromel-alumel thermocouple taped to the densimeter and is recorded during the density measurement after the propellant sample has reached thermal equilibrium with the environmental bath.

- (U) The apparatus used in the vapor pressure measurements consisted of a 10-milliliter, stainless-steel cylinder with immersion thermocouple, pressure transducer, and sample valve. The thermocouple, a chromel-alumel junction with stainless-steel sheath, was sealed into the cylinder with a swedge fitting, thus permitting direct measurement of the temperatures of the cylinder contents. The thermocouple was calibrated at the melting and boiling points of water. The 1000-psia pressure transducer was calibrated with a Heise gage over the temperature and pressure ranges of intended use.
- (U) During the measurements, a sufficient amount of ClF_3 was loaded into the vapor pressure apparatus to ensure the presence of some liquid at all times over the range considered. The bomb and contents were allowed to reach thermal equilibrium at selected temperatures, and the equilibrium vapor pressures were recorded. Constant temperatures above ambient were maintained by placing the entire apparatus in a Fisher Isotemp oven.
- (U) Density of Chlorine Trifluoride. With the variable volume capabilities of the Poole-Nyberg densimeter, it was possible to conduct measurements on propellant-grade ClF_3 over the temperature range of -22°C (-8°F) to -8°C (18°F) with one filling of the apparatus. The density data resulting from these measurements are presented in Table 1 with the chemical analysis of the propellant sample. These density data together with that obtained from previous experimental measurements (Ref. 1 and 2) were curve

fit by a least squares computer program over a temperature range of -22 C (-8 F) to 161 C (322 F), which resulted in the following equations:

$$\rho(\text{gm/cc}) = 4.924 - 2.517 \times 10^{-2} T_{(K)} + 7.3842 \times 10^{-5} T_{(K)}^2 - 8.215 \times 10^{-8} T_{(K)}^3 \quad (1)$$

$$\rho(\text{lb/ft}^3) = 121.360 - 0.1226 t_{(F)} + 2.127 \times 10^{-4} t_{(F)}^2 - 8.850 \times 10^{-7} t_{(F)}^3 \quad (2)$$

The standard deviations for these curve fits are 0.005 gm/cc and 0.31 lb/ft³, respectively. These equations are represented graphically in Fig. 2 and 3, respectively.

TABLE 1

EXPERIMENTAL DENSITY DATA FOR SATURATED LIQUID ClF₃*

Temperature, C	Observed Density, gm/cc	Calculated** Density, gm/cc	$\Delta \rho \times 10^3$, gm/cc
-22.53	1.958	1.9609	2.9
-20.15	1.949	1.9523	3.3
-16.18	1.940	1.9383	-1.7
-8.73	1.913	1.9128	-0.2

*Sample composition: ClF₃ — 99.5+ w/o
HF — 0.4 w/o
Noncondensables — trace

**From Eq. 1

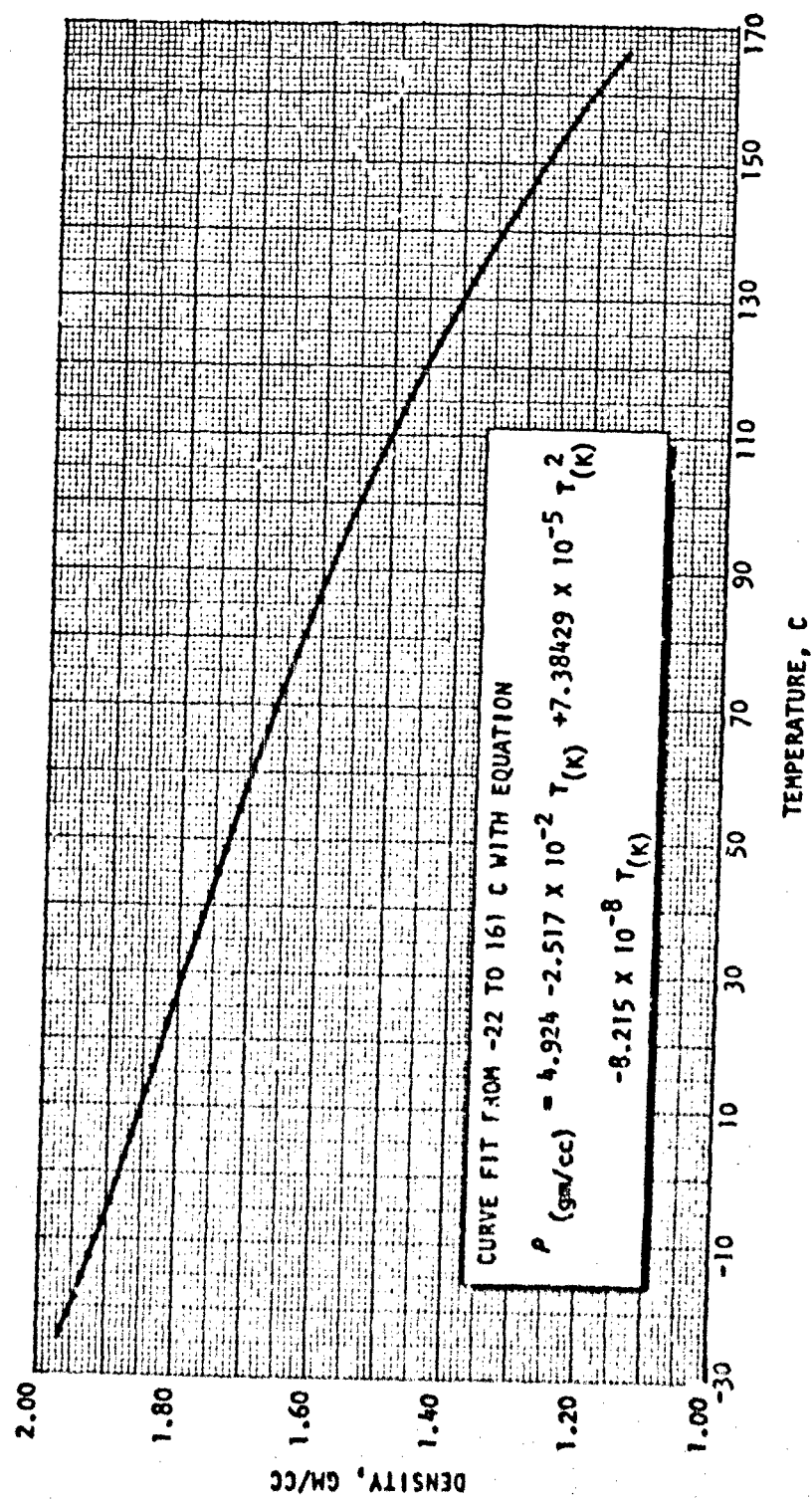


Figure 2. Density of Saturated Liquid Chlorine Trifluoride

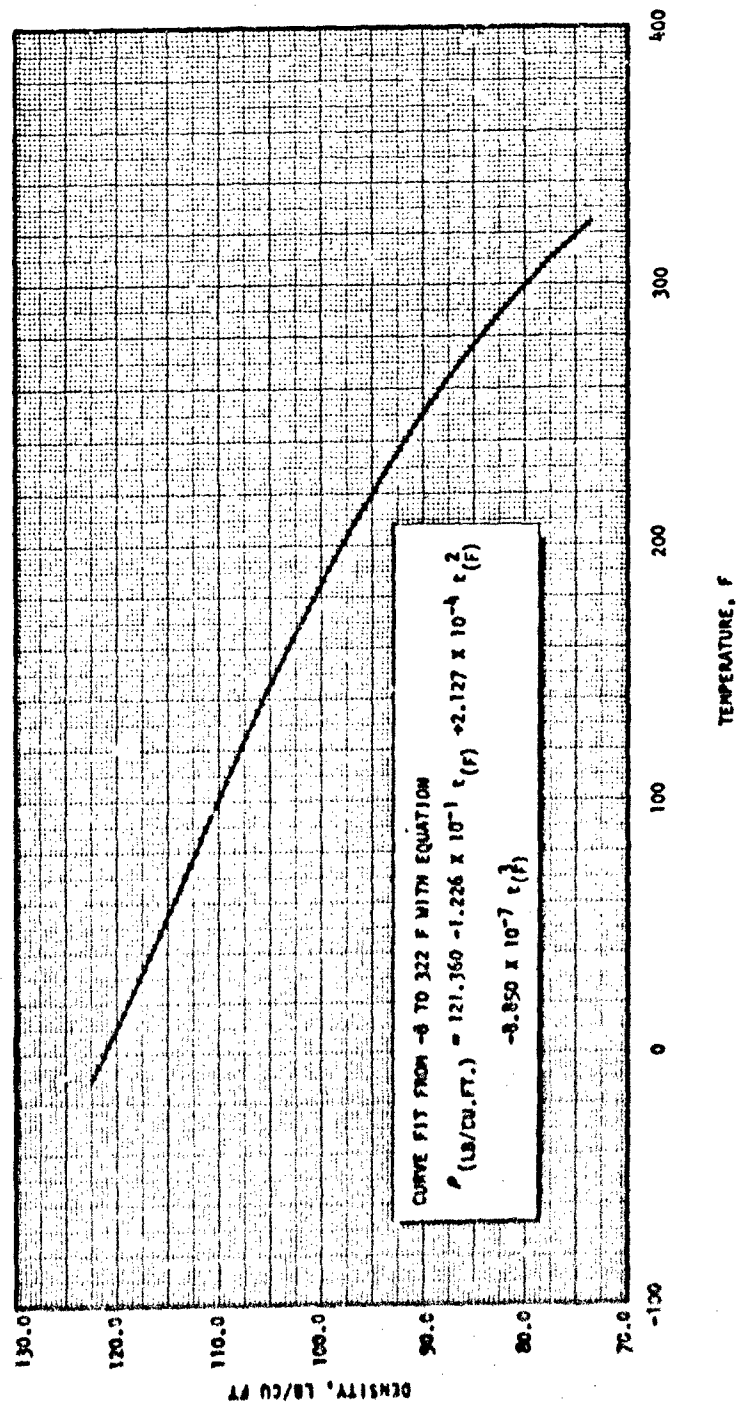


Figure 3. Density of Saturated Liquid Chlorine Trifluoride

(U) Vapor Pressure of Chlorine Trifluoride. The vapor pressure of propellant grade ClF_3 was measured over the temperature range of 42.9 C (109 F) to 147.6 C (298 F). The data obtained thus far are presented in Table 2 with the chemical analysis of the propellant sample. Upon completion of additional measurements in this temperature region and extension of the measurements to the critical temperature (355 F), all data, including that previously determined (Ref. 1 and 3), will be curve fit by a least squares computer program.

TABLE 2

EXPERIMENTAL VAPOR PRESSURE DATA FOR ClF_3 *

Temperature,		Pressure, psia
C	F	
42.9	109.2	45
61.1	142.0	78
74.6	166.3	114
93.1	199.6	182
111.3	232.3	274
131.4	268.5	408
147.6	297.7	560

*Sample composition: ClF_3 --99.5+ w/o
 HF--0.4 w/o
 Noncondensables--trace

Sonic Velocity (and Adiabatic Compressibility) Measurements

(U) Sonic velocity measurements were conducted in liquid chlorine trifluoride (ClF_3) and chlorine pentafluoride (ClF_5). These measurements covered a temperature range of -77 C (-107 F) to 69 C (156 F) and pressures of saturation, 500 and 1000 psia.

- (U) The experimental apparatus, which has been described previously in Ref. 1 and is illustrated in Fig. 4, is used to accurately measure the distance of test fluid through which sound waves of a known frequency travel. The interferometer, which is capable of withstanding pressures to 1000 psia and temperatures to 200 F, is constructed of type 347 stainless steel (which is compatible with most propellants of interest). The dial gage, which provides precise linear location data also enables the differentiation between the reflected signal (and its harmonics) and reflections from the metallic interferometer body. Displayed pips, from true reflection move on the oscilloscope as the reflector is moved, while spurious signals remain stationary.
- (U) The measurements are conducted by initiation of a 5-megacycle radio frequency signal from the pulsed oscillator, which is fed simultaneously to the oscilloscope and a quartz piezoelectric crystal (with 5-megacycle resonant frequency) attached to the bottom of the interferometer. The sound waves, emanating from the crystal, travel through the bottom of the interferometer, through a known distance of test liquid to a reflector, and then back to the crystal. The initial and reflected waves are displayed on the oscilloscope, thus allowing measurement of the time required for the ultrasonic waves to traverse the known distance of test fluid.
- (U) During the determinations, the interferometer was immersed in a constant-temperature bath and allowed to reach thermal equilibrium at a selected temperature level before a measurement was conducted. The equilibrium temperature was then measured using a chromel-alumel thermocouple (with a type 316 stainless-steel sheath) immersed in the test fluid.
- (U) The sonic velocity apparatus was calibrated over a temperature range of 0 C (32 F) to 74 C (165 F) at pressures of 14.7, 500, and 1000 psia, using distilled water and absolute methanol as test fluids. The data obtained from sonic velocity measurements in these fluids with this apparatus were compared with literature values for these fluids (Ref. 5, 6, and 7) to

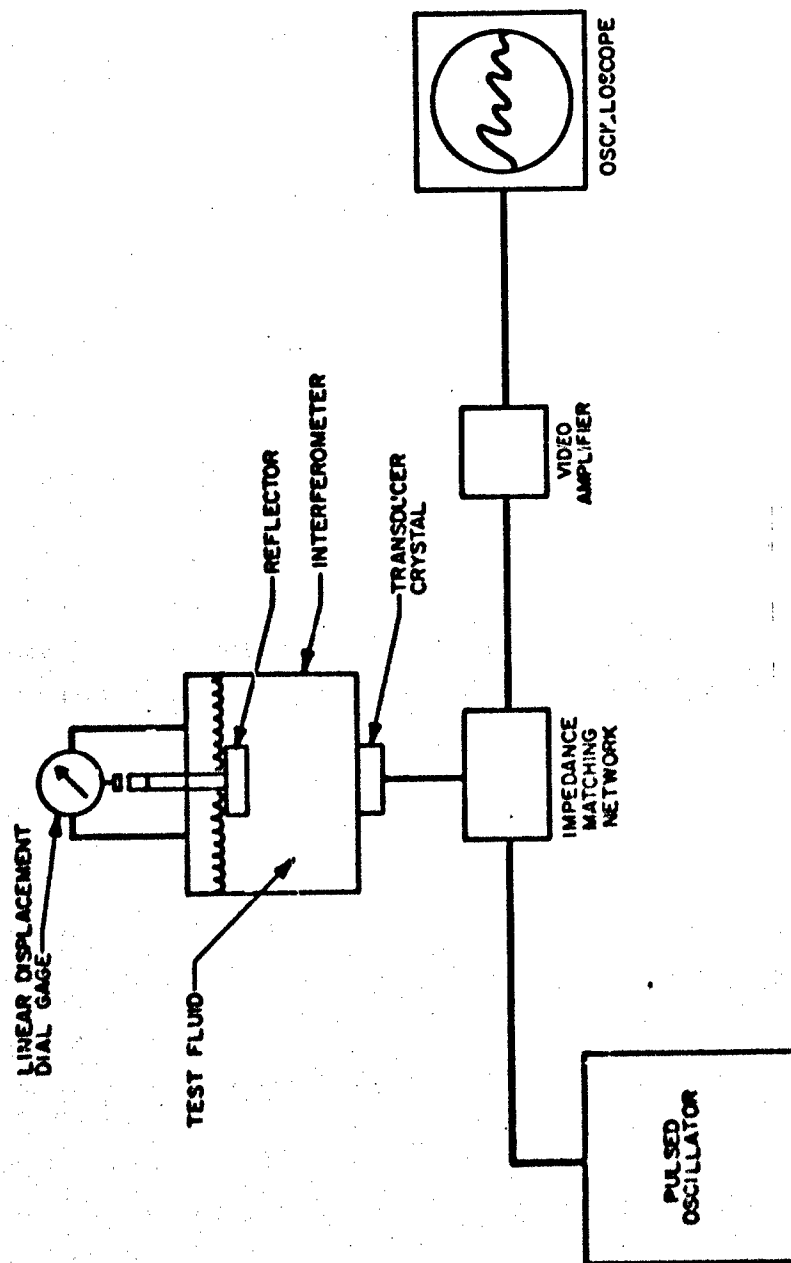


Figure 4. Sonic Velocity Apparatus

obtain a calibration factor. The agreement between the values obtained with this apparatus and those of the literature were within 1.5 percent with a precision of < 1 percent.

- (U) Using the resulting sonic velocity data and the liquid density, the adiabatic compressibility of the liquid was calculated from the relationship:

$$\beta_s = \frac{1}{\rho c^2} \quad (3)$$

where

β_s = adiabatic compressibility of the liquid

ρ = density of the liquid

c = velocity of sound in the liquid

- (U) Sonic Velocity of Chlorine Pentafluoride. The velocity of sound was measured in propellant-grade liquid ClF_5 under: (1) saturated liquid conditions over a temperature range of -77.1 C (-106.8 F) to 68.7 C (155.7 F); (2) total pressurization of 500 psia (using gaseous nitrogen) over a temperature range of -73.5 C (-100.3 F) to 69.2 C (156.6 F); and (3) total pressurization of 1000 psia (using gaseous nitrogen) over the temperature range -27 C (-16.6 F) to 16.8 C (62.2 F). The results of these measurements are presented in Table 3 with results of chemical analysis of the propellant sample.

- (U) The experimental data for the velocity of sound in ClF_5 under saturated liquid conditions were curve fit using a least squares computer program. The resulting equations, which represent the saturated liquid data from -77 to 69 C (-107 to 156 F) in both metric and English units are:

$$c(\text{m/sec}) = 1755 - 4.074 T(\text{K}) + 5.936 \times 10^{-4} T(\text{K})^2 \quad (4)$$

TABLE 3

EXPERIMENTAL SONIC VELOCITY DATA FOR LIQUID ClF_5 *

Temperature		Pressure	Sonic Velocity, m/sec		Δ , m/sec
C	F		Experimental	Calculated	
-77.1	-106.8	Saturation	979.0	978.6	0.4
-56.5	-69.7	Saturation	900.0	899.9	0.1
-31.0	-23.8	Saturation	801.0	803.1	-2.1
-19.6	-3.3	Saturation	757.0	760.0	-3.0
-6.0	22.2	Saturation	708.0	708.8	-0.8
-0.8	30.6	Saturation	692.0	689.3	2.7
0.4	32.7	Saturation	693.0	684.8	8.2
3.6	38.5	Saturation	677.0	672.8	4.2
8.3	46.9	Saturation	648.0	655.2	-7.2
15.1	59.2	Saturation	631.0	629.8	1.3
26.2	79.2	Saturation	586.0	588.3	-2.3
34.4	93.9	Saturation	555.0	557.8	-2.8
48.9	120.0	Saturation	504.0	504.0	0.0
68.7	155.7	Saturation	432.0	430.8	1.2
-73.5	-100.3	500 psia	951.0		
-54.2	-65.6	500 psia	883.0		
-27.9	-18.2	500 psia	785.0		
-18.2	-0.8	500 psia	754.0		
-1.9	29.6	500 psia	675.0		
4.8	40.6	500 psia	652.0		
11.0	51.8	500 psia	627.0		
22.4	72.3	500 psia	589.0		
25.7	78.3	500 psia	577.0		
33.6	92.5	500 psia	552.0		
43.5	110.3	500 psia	520.0		
54.7	130.5	500 psia	487.0		
69.2	156.6	500 psia	449.0		
-27.0	-16.6	1000 psia	760.0	--	--
-21.8	-7.2	1000 psia	745.0	--	--
-3.2	26.2	1000 psia	692.0	--	--
16.8	62.2	1000 psia	622.0	--	--

*Sample composition:

	ClF_5	ClF_3	HF	ClO_2
Prerun Analysis	98.5% w/o	0.8 w/o	0.6 w/o	<0.05 w/o
Postrun Analysis	98.8% w/o	0.5 w/o	0.6 w/o	<0.05 w/o

NOTE: Calculated sonic velocity values are those calculated at the measurement temperature using the appropriate curve-fit equation.

and

$$c(\text{ft/sec}) = 5758 - 7.426 T_{(R)} + 6.011 \times 10^{-4} T_{(R)}^2 \quad (5)$$

The standard deviations of these curve fits are 2.2 m/sec and 7.2 ft/sec, respectively. Graphical representations of these equations are presented in Fig. 5 and 6, respectively.

- (U) An anomaly was noted in the data resulting from the sonic velocity measurements in the pressurized liquid. Normally, the velocity of sound in a liquid at constant temperature has been found to increase with increasing pressure. In addition, the change in slope of a sonic velocity-temperature plot is relatively equivalent for various isobars over the same temperature range. The data at both 500 and 1000 psia indicate that at the lower temperatures investigated, the velocity of sound in ClF_5 was decreased as the saturated liquid was pressurized. However, as the temperature was increased, the saturated liquid plot and the isobars (which can be plotted from the data at 500 and 1000 psia) will eventually meet and reverse this condition.
- (U) Consideration of possible effects resulting from liquid solubility of the nitrogen gas, which was used for pressurization, offers a reasonable explanation of this anomaly. As gas content is increased in the liquid (this would accompany an increase in pressurant gas pressure), the velocity of sound will decrease. As temperature of the liquid is increased, the gas content (solubility) in the liquid should decrease. The combination of these two effects together with the assumed effect of pressure on sonic velocity in ClF_5 are probably responsible for the anomalous trends in the experimental observations.
- (U) To eliminate the influence of dissolved pressurant gas in the measurements, a bellows assembly could be used instead of a pressurant gas to apply pressure to the liquid sample. However, the small magnitude of the observed pressure effect (typically, a 0.5 percent difference in sonic velocity from

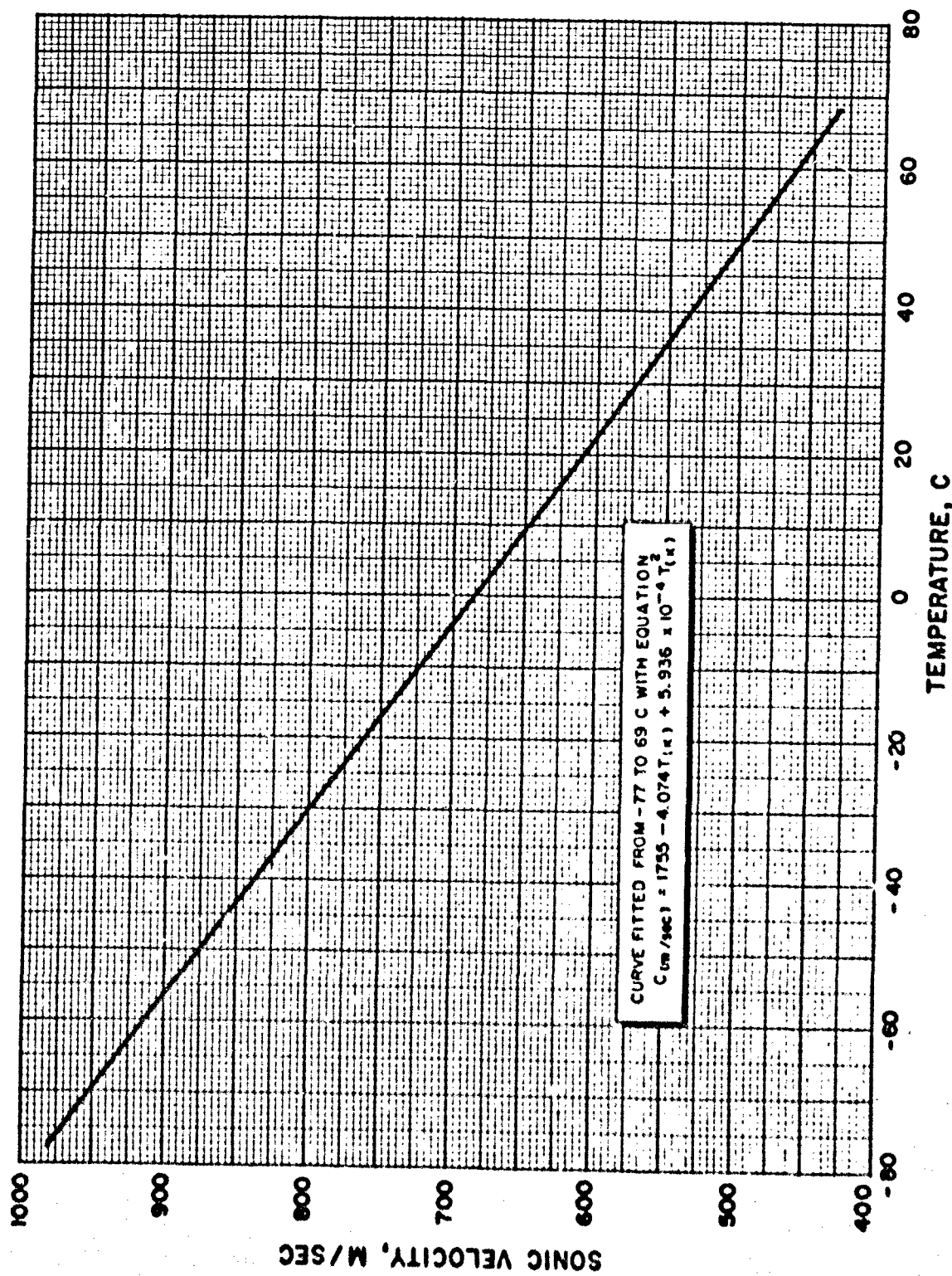


Figure 5. Sonic Velocity in Saturated Liquid Chlorine Pentafluoride

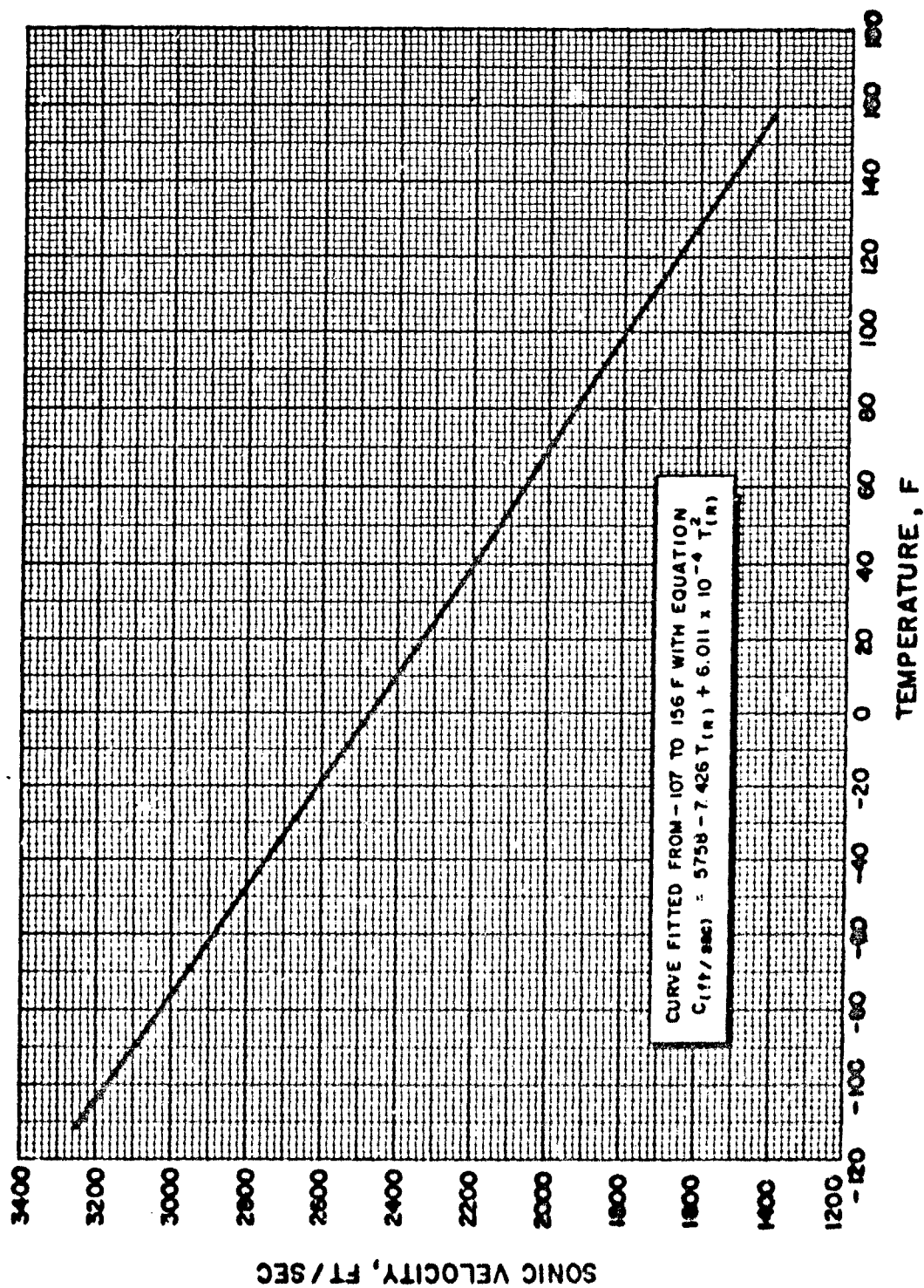


Figure 6. Sonic Velocity in Saturated Liquid Chlorine Pentafluoride

saturation to 500 psia) within the experimental conditions under consideration does not justify a modification of the apparatus at the present time. In addition, the effect of inert gas on physical properties should be considered because similar conditions are encountered in the design and development of propulsion systems.

(U) An experiment has been planned to demonstrate the effect on sonic velocity of a dissolved gas in a propellant. This effect, which will be characterized in both ClF_3 and ClF_5 , will be studied by determining the change in sonic velocity in a liquid sample as the sample is subjected over a period of time to a constant inert gas pressure at a preset temperature level.

(U) Adiabatic Compressibility of Chlorine Pentafluoride. The sonic velocity and density data (Ref. 8) of saturated liquid ClF_5 were used to compute adiabatic compressibilities according to Eq. 3. A least squares curve fit of the resulting calculations from -80 to 70 C (-112 to 158 F) established the following compressibility-temperature relationships for saturated liquid ClF_5 (in both metric and English units):

$$\begin{aligned} \beta_{(\text{atm}^{-1})} = & 1.1565 \times 10^{-4} + 1.3942 \times 10^{-6} t_{(\text{C})} + \\ & 1.2708 \times 10^{-8} t_{(\text{C})}^2 + 1.4680 \times 10^{-10} t_{(\text{C})}^3 + \\ & 9.6855 \times 10^{-13} t_{(\text{C})}^4 \end{aligned} \quad (6)$$

and

$$\begin{aligned} \beta_{(\text{psia}^{-1})} = & 6.4065 \times 10^{-6} + 4.0065 \times 10^{-8} t_{(\text{F})} + \\ & 1.4103 \times 10^{-10} t_{(\text{F})}^2 + 9.0915 \times 10^{-13} t_{(\text{F})}^3 + \\ & 6.2782 \times 10^{-15} t_{(\text{F})}^4 \end{aligned} \quad (7)$$

The standard deviation of these curve fits, which are graphically presented in Fig. 7 and 8, respectively, are $8.85 \times 10^{-7} \text{ atm}^{-1}$ and $6 \times 10^{-8} \text{ psia}^{-1}$, respectively.

- (U) Sonic Velocity of Chlorine Trifluoride. Sonic velocity measurements were conducted in propellant-grade liquid chlorine trifluoride, ClF_3 , under saturated liquid conditions over a temperature range of -59.7 to 74.5 C (-75.5 to 166.1 F), and under total pressures (using gaseous nitrogen) of 500 and 1000 psia over temperature ranges of -51.2 to 74.9 C (-60.2 to 166.8 F) and -49.5 to 34.1 C (-57.8 to 93.4 F), respectively. The results of these measurements and the chemical analysis of the propellant sample are presented in Table 4. A curve fit of the sonic velocity data obtained from measurements in saturated liquid ClF_3 resulted in the following equations.

$$c(\text{m/sec}) = 1951.8 - 3.7508 T(\text{K}) \quad (8)$$

and

$$c(\text{ft/sec}) = 6401.8 - 6.8348 T(\text{R}) \quad (9)$$

The standard deviations for these curve fits, illustrated in Fig. 9 and 10, respectively, are 2.9 m/sec and 9.4 ft/sec., respectively.

- (U) An anomaly, similar to that observed in the sonic velocity measurements in ClF_5 , appears in the data presented in Table 4. As noted in the previous discussion on ClF_5 , the velocity of sound in a liquid at constant temperature normally increases with increasing pressure, and the change in slope of a sonic velocity-temperature plot is approximately equivalent for various isobars over the same temperature range. Table 4 indicates erratic and conflicting results in some of the data obtained in the pressurization of the ClF_3 to 500 and 1000 psia. As noted previously, the probable cause of this anomaly is postulated as an effect of pressurization gas solubility in the liquid. This effect is currently being characterized.

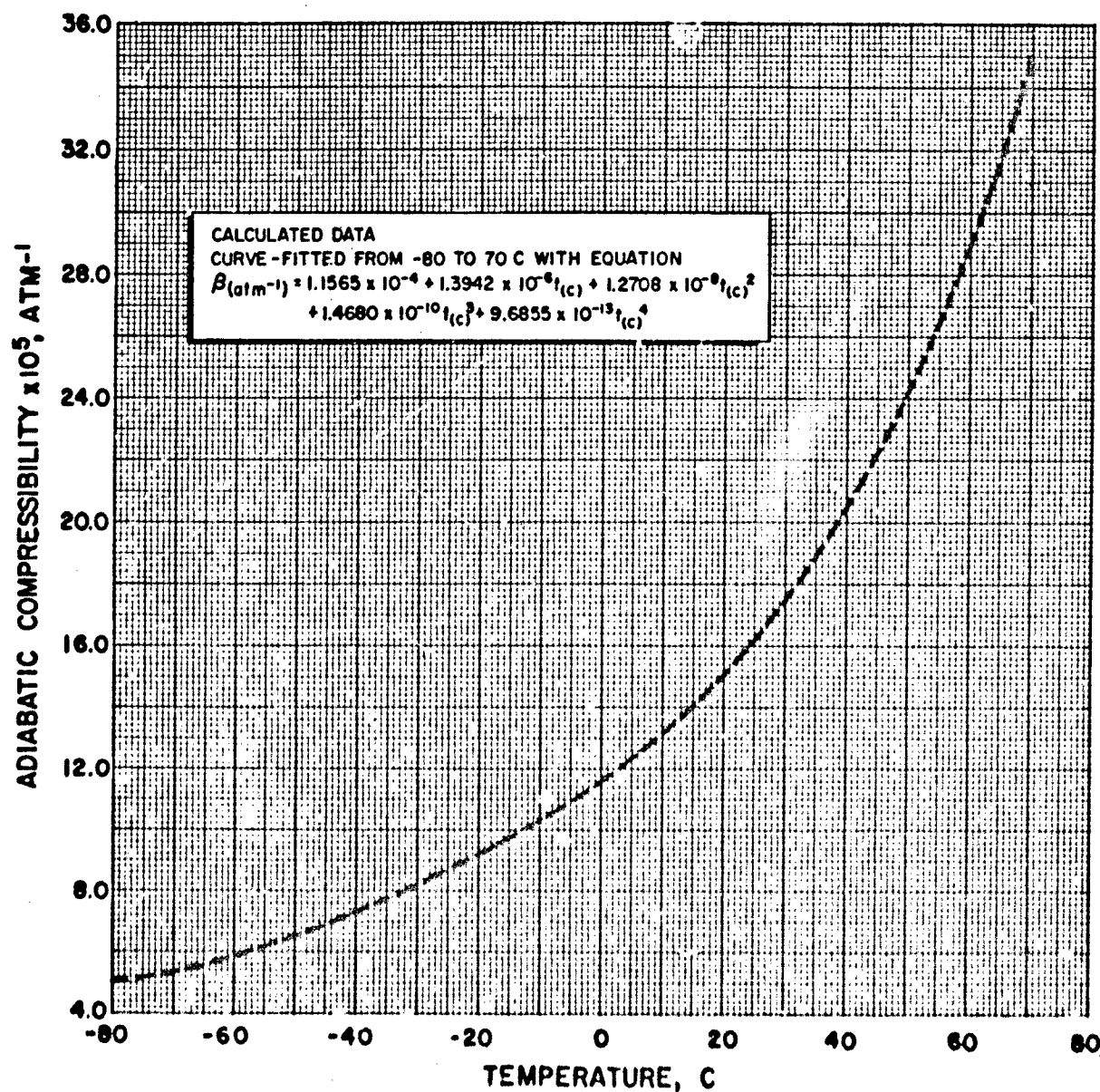


Figure 7. Adiabatic Compressibility of Saturated Liquid Chlorine Pentafluoride

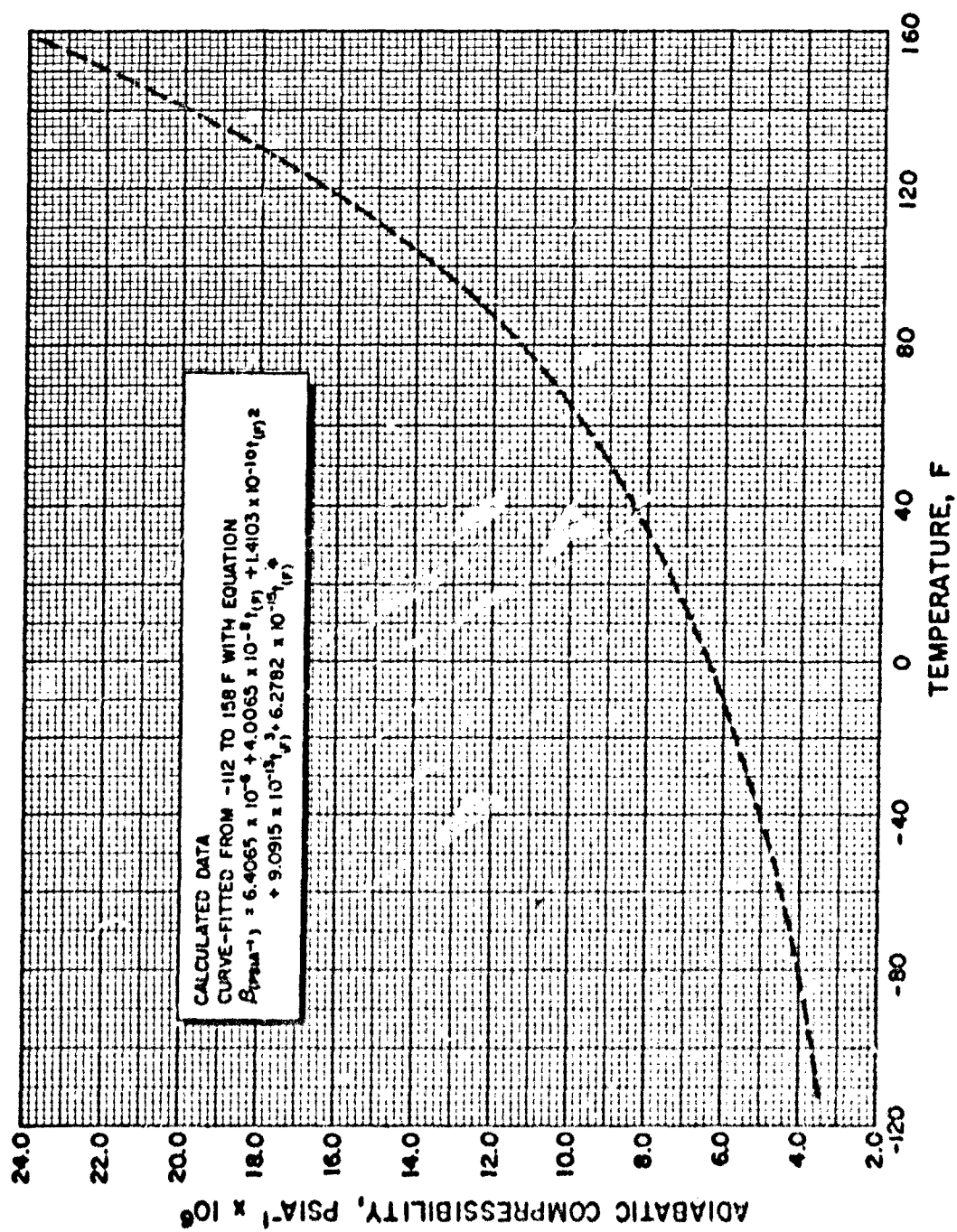


Figure 8. Adiabatic Compressibility of Saturated Liquid Chlorine Pentafluoride

TABLE 4

SONIC VELOCITY IN ClF_3^*

Temperature,		Pressure	Sonic Velocity, m/sec		Δ , m/sec
C	F		Experimental	Calculated	
-59.7	-75.5	Saturation	1157.4	1151.1	-6.3
-27.6	-17.7		1026.5	1030.7	4.3
-18.1	-0.6		990.5	995.1	4.6
-5.2	22.6		946.5	946.7	0.2
0.0	32.0		926.9	927.2	0.3
6.3	43.3		900.8	903.6	2.8
14.6	58.3		871.7	872.4	0.7
26.2	79.2		832.4	828.9	-3.5
36.2	97.2		794.0	791.4	-2.6
44.8	112.6		760.1	759.2	-0.9
55.6	132.1		718.2	718.7	0.5
63.5	146.3		683.5	689.0	-0.5
74.5	166.1		647.4	647.8	0.4
-51.2	-60.2	500 psia	1110.4	--	--
-27.3	-17.1		1018.7	--	--
-22.2	-8.0		1002.1	--	--
-15.3	4.5		977.8	--	--
-5.2	22.6		939.5	--	--
6.5	43.7		901.8	--	--
15.1	59.2		879.6	--	--
26.2	79.2		842.5	--	--
34.4	93.9		790.7	--	--
42.6	108.7		759.6	--	--
51.5	124.7		726.4	--	--
59.8	139.6		698.5	--	--
74.9	166.8		659.1	--	--
-49.9	-57.8	1000 psia	1114.0	--	--
20.2	68.4		851.6	--	--
34.1	93.4		802.9	--	--

*Sample composition:

	ClF_3	HF	ClO_2	Cl_2
Prerun Analysis	> 99.5 w/o	0.4 w/o	0.2 w/o	0.01 w/o
Postrun Analysis	> 99.1 w/o	0.5 w/o	0.3 w/o	0.01 w/o

NOTE: Calculated sonic velocity values are those calculated at the measurement temperature using the appropriate curve fit equation.

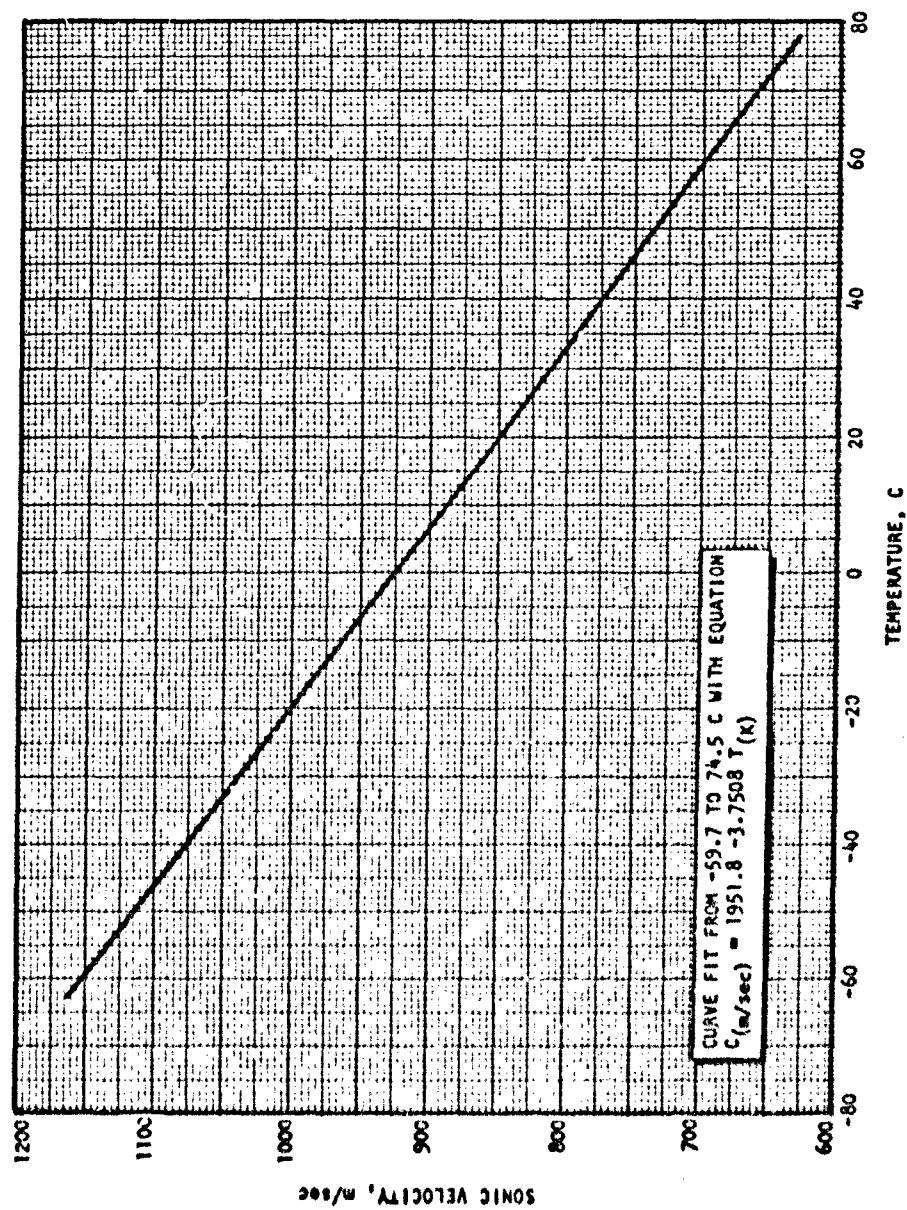


Figure 9. Sonic Velocity in Saturated Liquid Chlorine Trifluoride

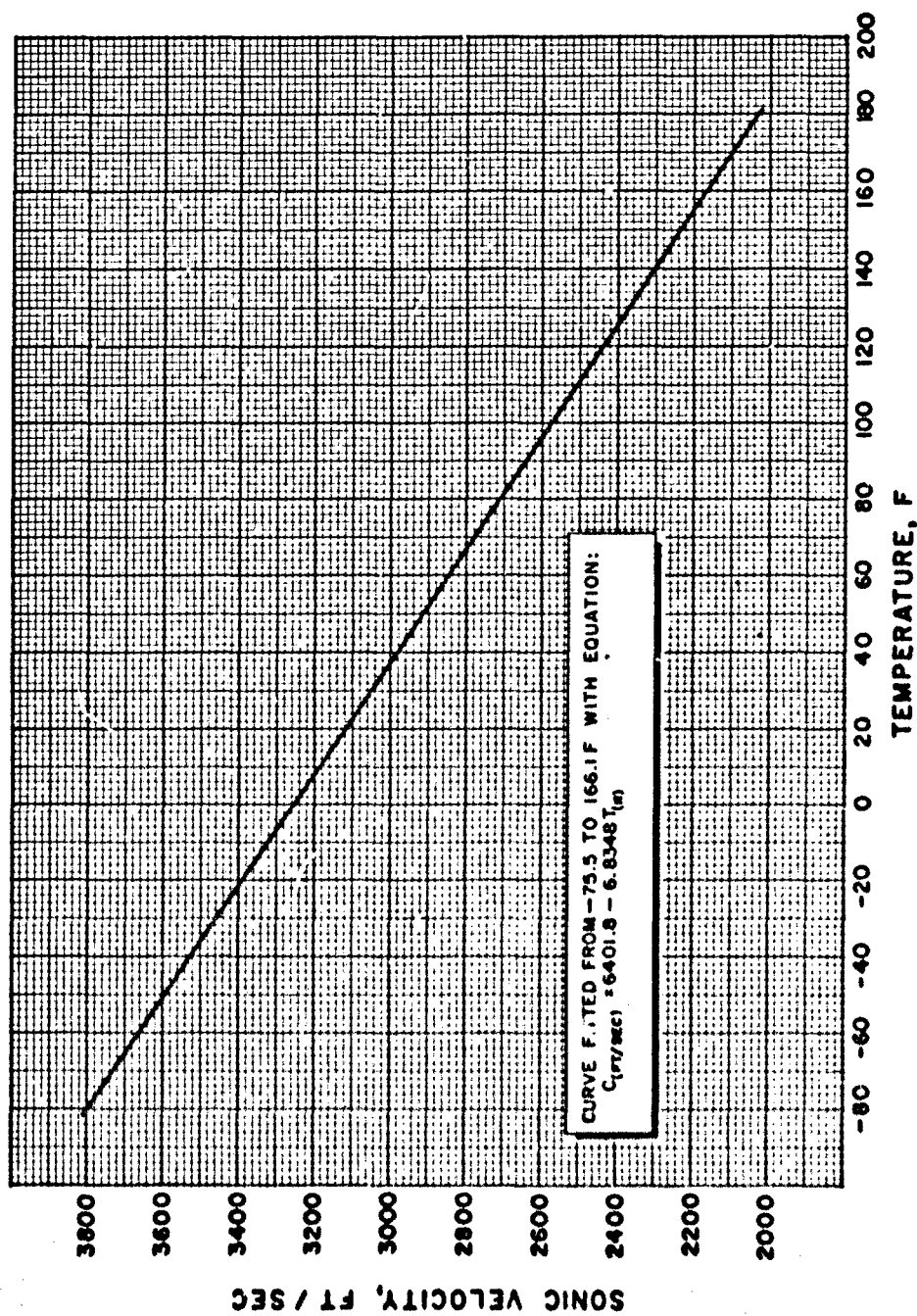


Figure 10. Sonic Velocity in Saturated Liquid Chlorine Trifluoride

- (U) Adiabatic Compressibility of Chlorine Trifluoride. Adiabatic compressibilities of ClF_3 were computed according to Eq. 3 using the sonic velocity data from the saturated liquid measurements and the saturated liquid density data noted previously. A least squares curve fit of the resulting calculations from -56 to 76 C (-69 F to 169 F) established the following compressibility-temperature relationships for saturated liquid ClF_3 (in both metric and English units):

$$\beta_{(\text{atm}^{-1})} = 6.2594 \times 10^{-3} + 6.1059 \times 10^{-5} t_{(\text{C})} + 3.7345 \times 10^{-7} t_{(\text{C})}^2 + 2.6649 \times 10^{-9} t_{(\text{C})}^3 + 2.1283 \times 10^{-11} t_{(\text{C})}^4 \quad (10)$$

$$\beta_{(\text{psia}^{-1})} = 3.5921 \times 10^{-4} + 1.8837 \times 10^{-6} t_{(\text{F})} + 5.7058 \times 10^{-9} t_{(\text{F})}^2 + 1.3434 \times 10^{-11} t_{(\text{F})}^3 + 1.3795 \times 10^{-13} t_{(\text{F})}^4 \quad (11)$$

The standard deviations of these curve fits, which are graphically presented in Fig. 11 and 12, are $4.5 \times 10^{-6} \text{ atm}^{-1}$ and $3.1 \times 10^{-7} \text{ psia}^{-1}$, respectively.

Inert Gas Solubility Measurements

- (U) Measurements of nitrogen gas solubility in propellant-grade chlorine pentafluoride are being conducted during the current program. In these determinations, the experimentally derived quantity is a differential solubility (the gas dissolved per unit mass of propellant and per unit pressure increase) measured at a particular temperature and pressure. This quantity can readily be integrated to give total (absolute) solubility.
- (U) A simplified schematic diagram of the apparatus, which has been previously described in detail (Ref. 1), is presented in Fig. 13. The inert gas is

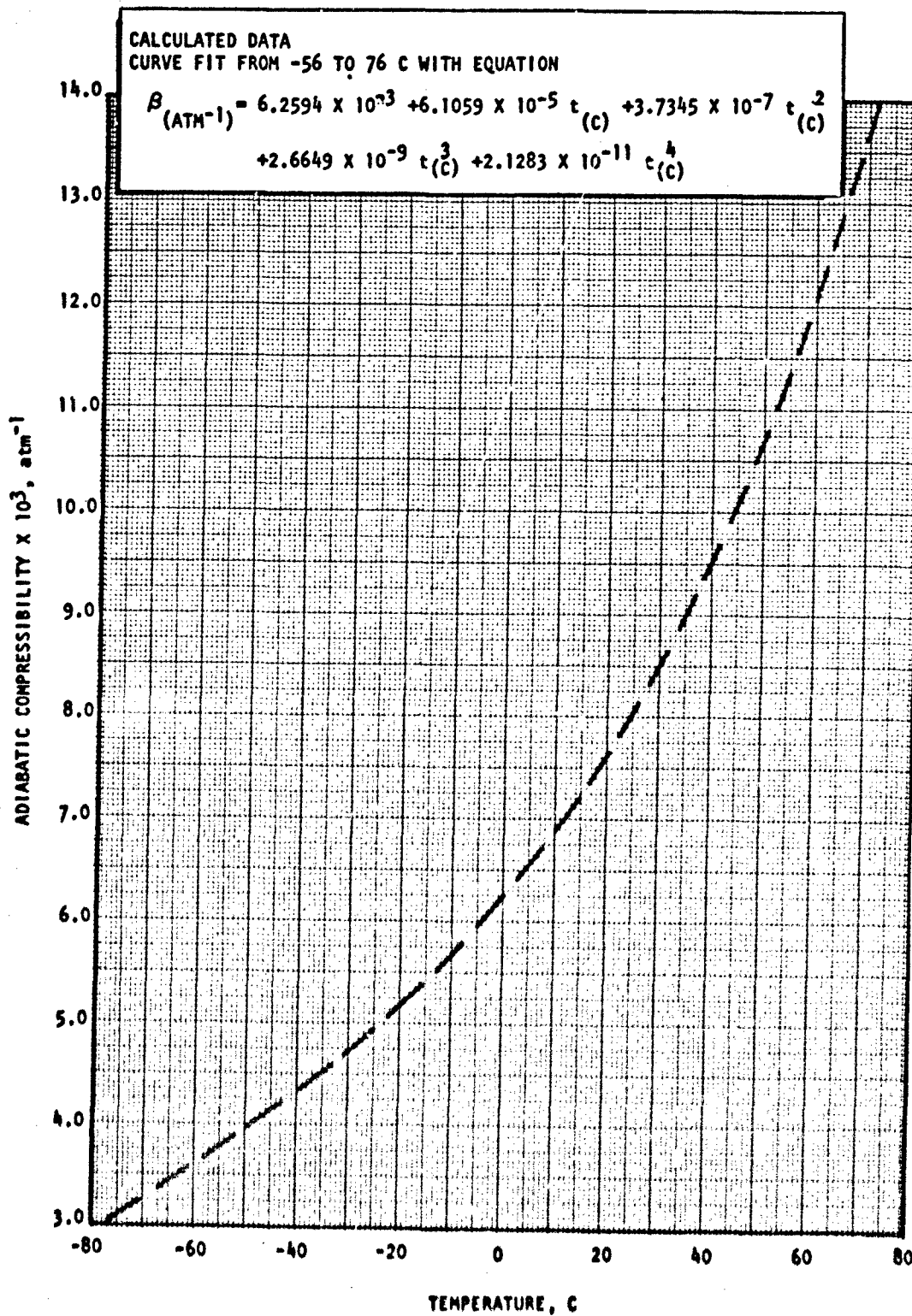


Figure 11. Adiabatic Compressibility of Saturated Liquid Chlorine Trifluoride

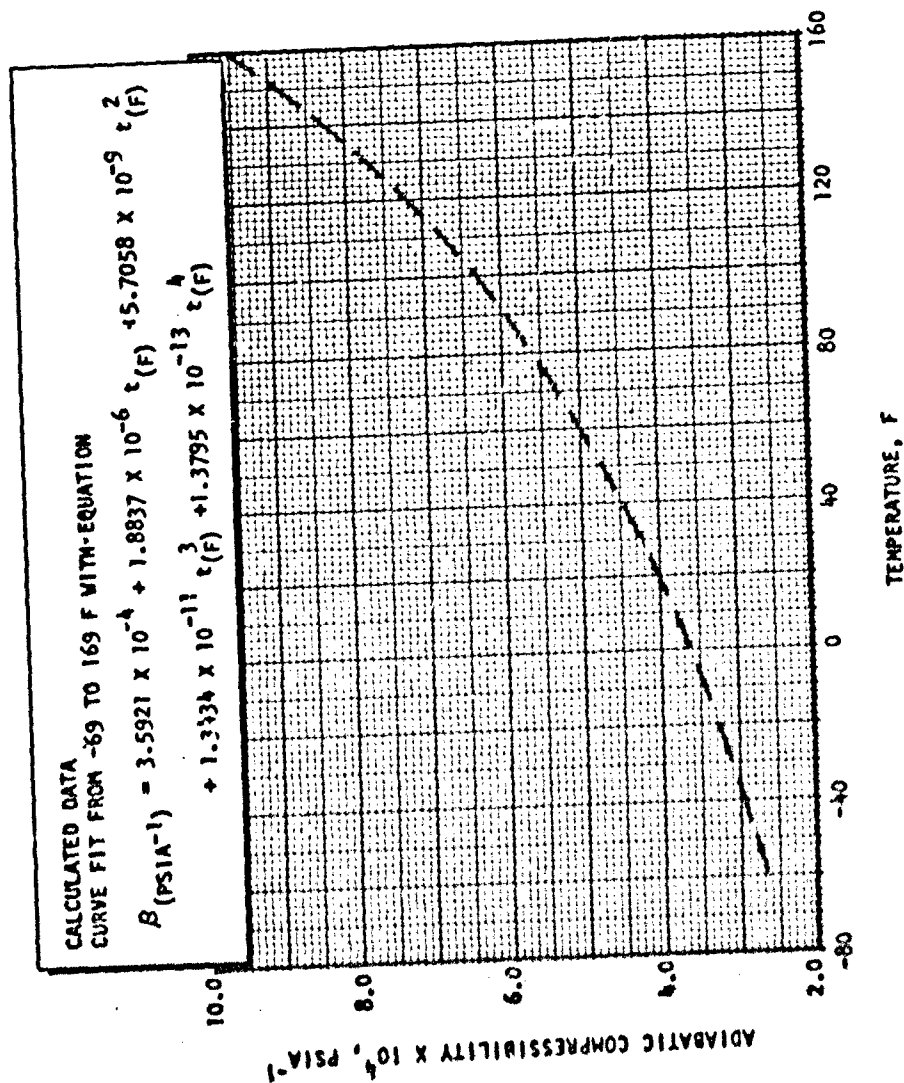


Figure 12. Adiabatic Compressibility of Saturated Liquid Chlorine Trifluoride

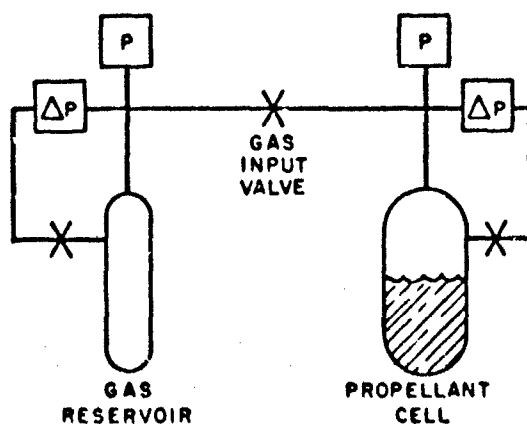


Figure 15. Simplified Schematic Diagram of Solubility Apparatus

introduced from a volume-calibrated gas reservoir into a volume-calibrated test tank, or propellant cell, which contains a known quantity of propellant. The volume of the gas absorbed at a known temperature and after agitation is calculated from pressure changes that occur in the system. These pressure changes are monitored by two precision differential pressure transducers. The entire apparatus, including both the propellant cell and the gas reservoir, is mounted within a thermostated enclosure, which maintains desired temperature during a solubility determination. The temperature conditioning enclosure is supported upon a rocking platform which is used to agitate the test solution in the propellant cell to attain equilibrium conditions.

- (U) At the beginning of a solubility determination, the propellant cell is charged with a measured quantity of liquid propellant, large enough to ensure that the cell will always contain a significant amount of liquid at the desired test temperature. The equilibrium cell pressure at the start of a test is then determined by the combined effects of propellant vapor pressure, inert gas content (e.g., total mass) in the cell, inert gas solubility, and relative volumes of ullage and liquid in the cell. The gas reservoir pressure is set at an adequately larger value than that required for supply during the solubility measurement.
- (U) The first step in a solubility determination consists of introducing gas from the reservoir into the cell ullage by temporarily opening the connecting input valve. The resulting pressure decrease in the reservoir and pressure increase in the cell are recorded by the corresponding differential pressure transducers which have controlling valves to isolate the initial pressures as reference pressures. The initial pseudo-equilibrium pressure in the cell (before inert gas can diffuse into the quiescent liquid to an appreciable extent) involves only ullage pressurization. A material balance between the reservoir gas and gas in the ullage can be written as follows:

$$V_G \left(\frac{\partial P}{\partial P} \right)_{T,G} (\Delta P)_G = V_U \left(\frac{\partial P}{\partial P} \right)_{T,U} (\Delta P)_{C,l} \quad (12)$$

where V_G is the reservoir volume which can contribute input gas; $(\partial\rho/\partial P)_{T,G}$ is the isothermal change of density with pressure for the pure reservoir gas at the test pressure; $(\Delta P)_G$ is the pressure drop recorded by the reservoir transducer; V_U is the ullage volume in the propellant cell; $(\partial\rho/\partial P)_{T,U}$ is the isothermal change in density with pressure for the inert gas in the vapor-gas ullage mixture at the test pressure, and $(\Delta P)_{C,1}$ is the initial pseudo-stable pressure increase in the cell.

- (U) The second step of a solubility determination is to agitate the liquid until solution equilibrium is established. Solution of inert gas causes the decline in cell pressure, and a lower differential pressure (than that obtained before agitation) is recorded by the cell pressure-transducer. Another material balance established between inert gas in the ullage and gas in solution can be written as follows:

$$V_U \left(\frac{\partial \rho}{\partial P} \right)_{T,U} [(\Delta P)_{C,1} - (\Delta P)_{C,2}] = sM (\Delta P)_{C,2} \quad (13)$$

where $(\Delta P)_{C,2}$ is the second (lower), equilibrium pressure increase in the propellant cell; s is the differential solubility of the inert gas in the propellant; and M is the mass of liquid propellant.

- (U) These material balances are combined to eliminate the ullage volume and gas compressibility, which are unknown quantities because the partial volume of solution and the P-V-T characteristics of the gas-vapor mixture in the ullage are unknown. (Experience to date verifies that these properties depart considerably from the ideals of a constant volume liquid and a perfect gas mixture.) To justify the cancellation of ullage properties, as well as the use of point values or averages for the density-pressure derivatives, the pressure range of a single test must be maintained reasonably low; the differential pressure transducers of the apparatus operate within a range of approximately 20 psi.

(U) The combination of Eq. (12) and (13) gives the following expression:

$$s = \frac{V_G}{M} \left(\frac{\partial \rho}{\partial P} \right)_{T,G} \frac{(\Delta P)_G \left[(\Delta P)_{C,1} - (\Delta P)_{C,2} \right]}{(\Delta P)_{C,1} (\Delta P)_{C,2}} \quad (14)$$

Thus, the establishment of the solubility requires a knowledge of the absolute volume of the gas reservoir tank, the mass of propellant in the cell, the density-pressure behavior of the pure inert gas, and the various transducer pressure readings.

(U) This experimental approach has proved to be generally sound. However, test experience revealed one experimental drawback; at low total pressures (i.e., large vapor/inert gas ratio in the ullage) the initial pressure equilibrium is not readily established following introduction of the gas into the ullage. Rather, the gas input induces some vapor condensation, and corresponding loss in cell pressure is only restored by a very slow (e.g., 20 minutes) transient buildup (controlled by either vapor-gas diffusion or heat transfer to the cell) under the necessary quiescent conditions. This slow equilibration is experimentally unacceptable, as it does not permit a definitive observation of the initial ullage equilibrium, unaffected by the process of solution. At higher total pressures (relative to the propellant vapor pressure), the effect diminishes and reliable observations are possible.

(U) Nitrogen Gas Solubility in Chlorine Pentafluoride. An initial set of data on nitrogen-chlorine pentafluoride solubility was obtained at 120 F. These data are presented in Table 5.

Because of the delayed equilibrium effect discussed previously, data could not be obtained at lower total pressure (the vapor pressure of ClF_5 is approximately 115 psia at 120 F). Because data acceptability is best judged by the precision of consecutive solubility measurements within a relatively small pressure range, the data points given in Table 5 are averages for several consecutive measurements within ± 10 percent or less. The scatter was largely associated with temperature fluctuations during a measurement.

TABLE 5

NITROGEN GAS SOLUBILITY IN CHLORINE PENTAFLUORIDE

Temperature, F	Total Pressure, psia	Solubility $\times 10^{-5}$, lb N_2 /lb ClF_5 -psi
120	400	2.58
120	610	2.94
120	780	3.26
120	910	3.28
90	350	2.35

- (U) Before attempting further measurements, the experimental arrangement was modified to provide the most direct possible input of gas to the propellant cell ullage (as permitted by the design of the cell) to minimize condensation and slow diffusion mixing within restricted line volumes. (The propellant cell necessarily has line connections for the input gas, differential pressure transducer, total pressure transducer, and safety burst diaphragm.) Other modifications included relocation of the transducer monitoring recorders to permit closer control of the input rate by the operator and the incorporation of more stable thermal control.
- (U) These modifications resulted in higher precision in the high-pressure region, but negligible improvement with regard to the initial equilibration problem. Attempted measurements at low pressure and temperature between 90 and 180 F yielded no useful results. Tentative experimental procedures involving slow and/or pulsed gas inputs with small thermal and mechanical shocks were unsuccessful.
- (U) Following these modifications, a reliable solubility data point (three consecutive measurements ± 1 percent) was obtained at 90 F where the vapor pressure of ClF_5 is approximately 72 psia. This point is shown as the last data point in Table 5. This test sequence was not extended further

to experimentally favorable high nitrogen contents because of corrosion failure of the valve controlling the propellant cell differential-pressure transducer. Testing will resume upon replacement of this component.

Specific Heat Measurements

- (U) The specific heats of UDMH, N_2H_4 -UDMH (50-50) and MHF-3 were measured in the calorimeter developed previously under Contract AF04(611)-9563 (Ref. 8) and further utilized under Contract AF04(611)-10546 (Ref. 1). The calorimeter consists of an outer copper jacket surrounding a small dewar vessel which is suspended by nylon cord from a copper ring attached to the top plate of the calorimeter. A sample container is positioned in the center of the dewar vessel and is isolated from it by several small pieces of foamed polystyrene insulation. A vacuum of 10^{-5} mm Hg is maintained in the calorimeter system to control conduction heat losses, and the silvered walls of the dewar vessel reduce radiative heat transfers. The calorimeter is completely immersed in a liquid bath medium.
- (U) Four cylindrical sample containers with volumes from 12 to 15 cc were used during the present contract. They were equipped with a filling tube extending from one end, and a heater winding of No. 30 B&S gage constantan wire. The windings were coated with glyptal and covered with copper foil to reduce the heat loss by radiation. A copper-constantan thermocouple was used for measuring the temperature. Significant differences among the four sample containers are described in Table 6.
- (U) A diagram of the electrical circuitry is shown in Fig. 14; the energy supplied to the heaters is accurately determined by means of a K-3 potentiometer in line with a galvanometer. The output from the sample container thermocouple is recorded by means of another K-3 potentiometer in conjunction with a d-c microvolt amplifier and recorder.

TABLE 6

DESCRIPTION OF SAMPLE CONTAINERS

Container No.	Material	Equilibration Fins	Thermocouple Position
I	Copper, Pb-Sn Solder	Yes	Exterior
II	Aluminum	Yes	Exterior
III	Copper, Gold Plated, Pb-Sn Solder	Yes	Well
IV	Copper, Unplated, Ag Solder	No	Well

(U) When previous measurements with this apparatus indicated discrepancies in the recorded data (Ref. 1), a company-initiated program was undertaken to locate and define the measurement errors. This effort was completed prior to the start of this report period and led to certain modifications which were incorporated into the apparatus. In the original design (Ref. 1), the calorimeter included an electrically heated copper shield surrounding the sample container. Because the source of the error in the thermocouple electromotive force reading (Ref. 1) was located in the heat shield circuitry, the heat shield was replaced with a small dewar vessel (to reduce temperature drift). The electrical leads attached to the sample container windings were modified to decrease the IR drop across them. The resistance across the volt box was increased, reducing that current loss (Ref. 1) from ~ 1 percent to less than 0.1 percent. A further modification of the general calorimeter system consisted of completely immersing the calorimeter in the bath, thereby reducing the time required for the lid to reach a thermal steady state.

(U) Calibration and Testing of Apparatus. Prior to each series of measurements, the sample container to be used was calibrated empty at two or more temperatures so that its heat capacity could be subtracted from the measured values. Verification tests also were occasionally conducted employing

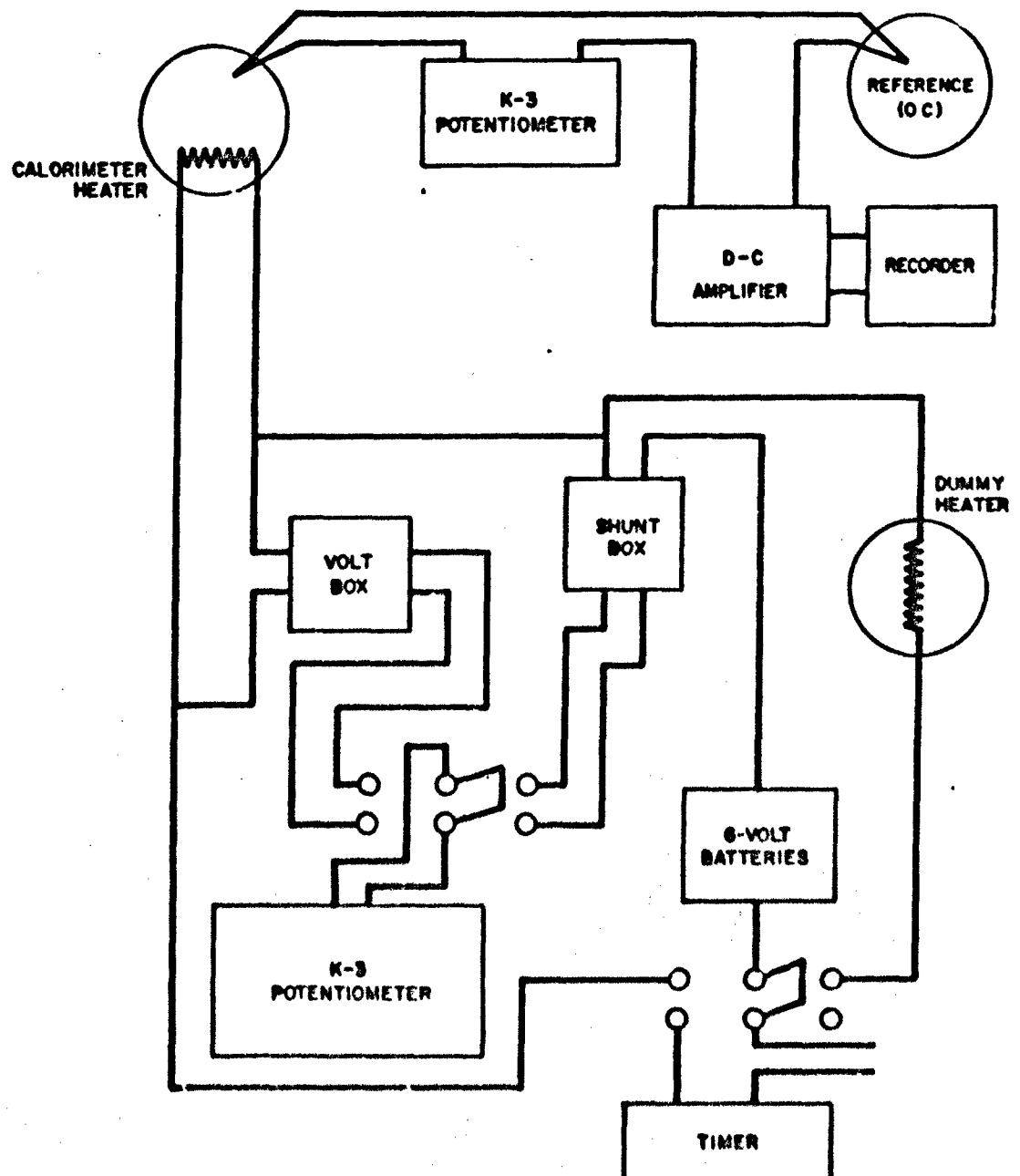


Figure 14. Block Diagram of Circuit Used in Specific Heat Measurements

liquids with known specific heats. This ensured that the various modifications did not adversely affect the accuracy of the measurements. The calibration and testing of the various modifications of the apparatus are detailed in the following paragraphs.

- (U) Sample Container No. I. After the previously mentioned circuitry modifications had been completed (but with the calorimeter not completely immersed), measurements were conducted with sample container No. I to determine the specific heat of spectrograde methanol. The values obtained agreed within 1 percent of those reported in the literature. In addition, the effects of variables such as the cooling or warming rates (negligible with maintenance of reasonable rates), power input rate (best results when the power input was kept constant from calibration to sample run), and heating period (no noticeable effect) were determined.
- (U) Sample Container No. II. An empty calibration run with an aluminum sample container exhibited good precision; but measurements of the specific heat of water yielded high, scattered results. Because the new circuitry had been checked by the methanol run, the discrepancies in the water results were thought to be related to the sample container itself.
- (U) Sample Container No. III. A copper sample container was fabricated with all internal parts gold-plated to make it more adaptable for use with a wide variety of liquids including the hydrazine-type fuels. A new feature of this sample container was its thermocouple well which is a centrally located cylinder within the container. Thin, copper disks, spaced along the thermocouple well in contact with the outer wall of the container, aided thermal equilibration.
- (U) Using this sample container, the specific heat of spectrograde methanol (dry-box loaded) was measured over the temperature range from -23 to 60 C.

The empty sample container was calibrated at room temperature and at 51 C. Analysis of these data resulted in a consistent linear heat capacity-temperature calibration curve for the empty calorimeter and established that the apparatus would provide the desired accuracy. Because the calibration curve is linear, a satisfactory sample container calibration requires only determination of the heat capacity of the empty container at two sufficiently separated temperatures.

- (U) During specific heat measurements on the N_2H_4 -UDMH (50-50) fuel blend, this sample container developed leaks in the crimped section of the filling tube, which was capped with Pb-Sn solder. Tests indicated that a good (helium-leakproof) cold-weld copper crimp can be made, but additional studies demonstrated that hydrazine fuels eventually leak through the crimp and that Pb-Sn solder possesses only a limited effectiveness in sealing against hydrazine-type fuel leaks. This problem was finally solved with the use of a silver solder capping on the crimp. However, the gold-plated sample container had been fabricated with Pb-Sn solder and during subsequent testing, leaks also developed in the weld around its bottom cap.

- (U) Sample Container No. IV. A new unplated copper sample container was fabricated with silver solder welds (sample container No. IV). After further apparatus modification (complete immersion), it was necessary to reconfirm the accuracy of the equipment and technique employed; a verification run was conducted with hydrazine (98.8 w/o N_2H_4 , 0.5 w/o NH_3 , 0.7 w/o H_2O). Three measurements of its specific heat were made at 19 C. The average of the results agreed exactly with the literature value (Ref. 9); the average deviation between the three measured values was approximately 1 percent.

- (U) Compatibility. Pressure build-up tests with containers No. III and IV indicated that their compatibility with N_2H_4 -type fuels was satisfactory at room temperature for the period of time involved in the specific heat measurements. Only 4×10^{-3} percent decomposition of N_2H_4 occurred in the unplated sample container, No. IV, over a 5-day period.

(U) Calculation of Results. The amount of electrical energy input, the resultant temperature rise (ΔT), and the saturation specific heat (C_s) are normally calculated using the equations contained in Ref. 1. In some runs, ΔT was calculated using an alternative method employing a series of equations derived by Regnault and Pfaundler (Ref. 10):

$$\Delta T = (T_2 - T_1) + k (A) \quad (15)$$

and

$$k = \frac{V_1 - V_2}{T_2 - T_1} \quad (16)$$

where

V_1 and V_2 = the linear rate of temperature change preceding and following the heating period, respectively.

T_1 and T_2 = the temperatures at which the extrapolated linear portion of the heating curve intersects the extrapolated ante- and post-period temperature drift curves, respectively.

k = Newtonian cooling constant

A = area beneath the extrapolated heating period of the experimental time-temperature curve bounded by the zero-drift temperature (T_0)

T_0 = zero drift temperature which is the apparent temperature of the heat sink as calculated by:

$$T_0 = \frac{V_1 T_2 - V_2 T_1}{V_1 - V_2} \quad (17)$$

The method of Regnault and Pfaundler provides further insight into the interpretation of the results of individual experiments. For example, a variable zero-drift temperature and a considerable temperature difference between T_0 and T_1 produce a negative Newtonian cooling constant, which is sufficient reason to eliminate a given run. Such a negative cooling constant suggests that the temperature of the system drifted during the measurement.

The complete immersion of the calorimeter (a modification suggested by the results of this method of data treatment) has resulted in considerably reduced temperature drift rates.

- (U) Specific Heat of UDMH. The results of Aston et al. for the specific heat of UDMH (Ref. 11) from the melting point (-57 to 25 C)(-71 to 77 F) fit the linear expression:

$$C_p \text{ (cal/gm-C)} = 0.640 + 0.00057 t_{(C)} \quad (18)$$

The aluminum sample container (No. II) was used to extend these measurements to 70.5 C (159 F). The results obtained are presented in Table 7. These values, however, averaged 4 to 5 percent greater than the values given by Eq. 18, even at the overlapping temperature range in the area of 25 C. This and the high values obtained for the specific heat of water in this sample container suggests that aluminum is not a satisfactory material for use in these studies.

- (U) Additional determinations were conducted on UDMH using the unplated copper sample container (No. IV) as a verification run prior to measurement of the specific heat of N_2H_4 -UDMH (50-50). These results with UDMH, also presented in Table 7, had an average deviation of 1.4 percent and averaged 1.8 percent above the values given by Eq. 18. The discrepancies in these various sets of data are presently being analyzed and conclusions will be presented in future reports under the contract.

- (U) Specific Heat of N_2H_4 . The results of Scott for the specific heat of hydrazine (Ref. 9) from 2 to 67 C (36 to 153 F) fit the following linear expression:

$$C_p \text{ (cal/gm-C)} = 0.726 + 0.00049 t_{(C)} \quad (19)$$

TABLE 7

EXPERIMENTAL SATURATED SPECIFIC HEAT OF UDMH

Average Temperature,		Specific Heat, cal/gm-C
C	F	
1.7	35.1	0.657 ^a
2.8	37.0	0.655 ^a
8.9	48.0	0.650 ^a
10.0	50.0	0.673 ^a
16.0	60.8	0.652 ^a
17.1	62.7	0.672 ^a
22.4	72.3	0.670 ^b
23.4	74.1	0.689 ^b
24.4	75.9	0.689 ^b
29.6	85.3	0.701 ^b
30.1	86.2	0.675 ^b
31.1	88.0	0.690 ^b
35.4	95.7	0.693 ^b
36.3	97.3	0.696 ^b
44.2	111.6	0.693 ^b
45.1	113.2	0.705 ^b
50.8	123.4	0.693 ^b
51.9	125.4	0.701 ^b
52.8	127.4	0.695 ^b
53.6	128.8	0.698 ^b
59.8	139.6	0.694 ^b
60.4	140.7	0.704 ^b
64.0	147.2	0.704 ^b
65.0	149.0	0.704 ^b
68.9	156.0	0.716 ^b
69.8	157.6	0.684 ^b
70.5	158.9	0.704 ^b

- a. Copper sample container (No. IV); analysis of UDMH--98.8 w/o UDMH, 0.8 w/o H₂O, 0.4t w/o other soluble impurities (with traces of NH₃ and (CH₃)₂ NH)
- b. Aluminum sample container (No. II); analysis of UDMH--99.4 w/o UDMH, 0.3 w/o H₂O, 0.2 w/o (CH₃)₂ NH, 0.1t w/o other soluble impurities

Three verification runs made in the unplated copper calorimeter (No. IV) yielded the following values for the specific heat of N_2H_4 :

Temperature, C (F)	C_p , cal/gm-C
17.7 (64)	0.742
18.8 (66)	0.737
19.8 (68)	0.727

The average of these three results agrees exactly with the value given by Eq. 19.

- (U) Specific Heat of N_2H_4 -UDMH (50-50). Specific heat measurements were conducted on propellant-grade N_2H_4 -UDMH (50-50) over the temperature range from 2.2 to 49.2 C (36 to 120 F). The results are listed in Table 8 and shown in Fig. 15. Results obtained in both the plated and unplated copper calorimeters, (No. III and IV) agreed quite well. The experimental specific heat data were curve fit to the following linear expression over the temperature range from 2 to 38 C (36 to 100 F):

$$C_p \text{ (cal/gm-C)} = 0.715 + 0.00047 t \text{ (C)} \quad (20)$$

The average deviation of the data from this curve is 0.8 percent. Based on two data points, the specific heat of this fuel blend appears to increase nonlinearly above 38 C.

- (U) The measured specific heat for N_2H_4 -UDMH (50-50) is approximately 5 percent greater than the previously reported calculated values (Ref. 12). The previous calculations were based on the specific heats of the individual components (Eq. 18 and 19) and an assumption of ideality between the two components. The deviation of the actual specific heat of this blend from that of an ideal solution appears reasonable. This deviation is well established in the present study because both of the components and the mixture were run in the same calorimeter.

TABLE 8

EXPERIMENTAL SATURATED SPECIFIC HEAT OF 50 W/O N_2H_4 -
50 W/O UDMH FUEL BLEND*

Average Temperature,		Specific Heat, cal/gm-C
C	F	
2.2	36.0	0.723 ^a
3.1	37.6	0.710 ^a
4.6	40.3	0.715 ^b
5.8	42.4	0.726 ^b
10.7	51.3	0.723 ^b
11.8	53.2	0.718 ^b
16.6	61.9	0.727 ^b
17.7	63.7	0.727 ^b
22.8	73.0	0.725 ^a
23.2	73.8	0.739 ^a
24.6	76.3	0.734 ^b
27.8	82.0	0.725 ^a
28.9	84.0	0.735 ^a
29.7	85.5	0.732 ^a
35.5	95.9	0.800 ^{a,c}
36.5	97.7	0.850 ^{a,c}
36.9	98.4	0.741 ^a
37.7	99.9	0.721 ^a
48.3	118.9	0.764 ^a
49.2	120.4	0.754 ^a

*Sample composition: 51.3 w/o N_2H_4 , 48.3 w/o UDMH, 0.4 w/o H_2O and other soluble impurities.

- a. Gold-plated copper sample container (No. III)
- b. Copper sample container (No. IV)
- c. Values appear to be erroneous; however, no experimental reason exists for omission.

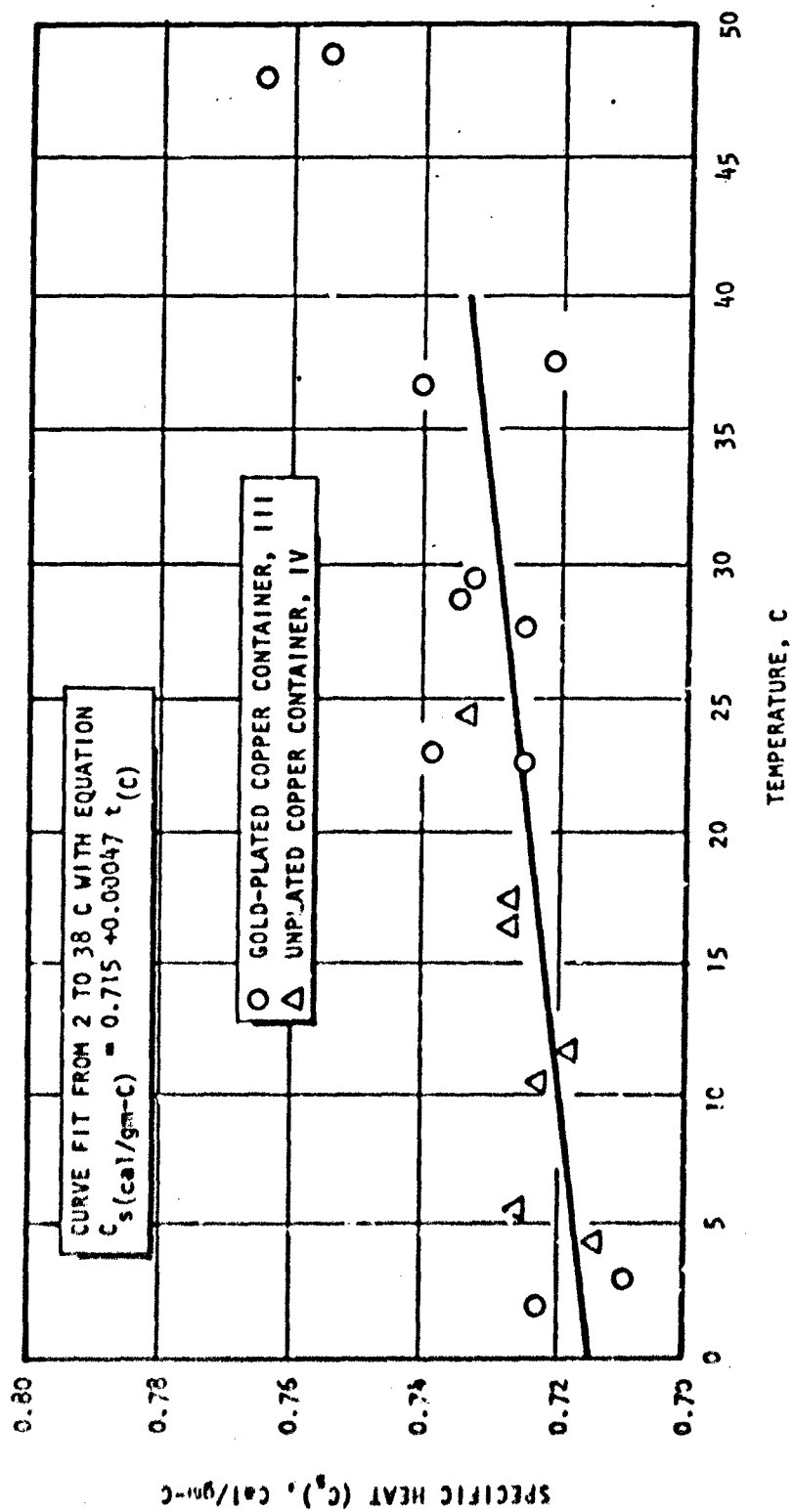


Figure 15. Saturated Specific Heat of N_2H_4 -UDMH (50-50) Fuel Blend

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- (U) The only experimental data reported previously by others for this fuel blend, 0.692 cal/gm-C at 27.3 C and 0.698 cal/gm-C at 38.8 C (Ref. 13), are also approximately 5 percent lower than the results obtained during the present study (Eq. 20). However, this earlier experimental work was conducted with a method-of-mixtures calorimeter, which has a normal maximum accuracy of ± 5 percent. An error of at least 5 percent in these earlier data would be expected because a single dewar vessel was used instead of the customary two (for the blank and the sample), identical heat losses were assumed in all cases, the effects of vaporization during opening were ignored, and a constant-temperature container was not used for the calorimeter dewar vessel.
- (C) Specific Heat of MIF-3. Specific heat measurements were initiated on a sample of MIF-3 (nominal composition: 86 w/o N_2H_4 -14 w/o $CH_3N_2H_3$) over a temperature range of -47.8 to 24.9 C (-54 to 77 F). However, the resulting data (Table 9) exhibited as much as 5 percent scatter in the worst cases. Because apparatus calibration data (obtained from subsequent specific heat measurements on the empty sample container) appear to have good precision, and excellent reproducible results have been obtained from the calorimeter during recent months in measurements on similar materials, the integrity of this set of measurements on MIF-3 is suspect. The reason for the poor precision and the apparently anomalous results is under investigation, and there is an indication that the problem is in the electrical equipment in the calorimeter circuit.

Thermal Conductivity

- (U) The thermal conductivities of three propellants, unsymmetrical dimethylhydrazine (i.e., UDMH or $(CH_3)_2N_2H_2$), MIF-3, and MIF-5, were measured thus far in this program. The apparatus used for obtaining thermal conductivity data was a steady-state, concentric cylinder conductivity cell, which was used during the preceding contractual effort (Ref. 1) to measure the thermal conductivity of N_2H_4 - $(CH_3)_2N_2H_2$ (50-50) and monomethylhydrazine (i.e., MBI or $CH_3N_2H_3$).

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TABLE 9

SATURATED SPECIFIC HEAT OF MHF-3 (PRELIMINARY DATA)

Temperature		Specific Heat, cal/gm-C
C	F	
-47.8	-54.0	0.726
-46.6	-51.9	0.706
-40.9	-41.6	0.703
-39.6	-39.3	0.706
-34.5	-30.1	0.705
-33.4	-28.1	0.732
-27.8	-18.0	0.721
-26.6	-15.9	0.717
-22.1	-7.8	0.684
-21.1	-6.0	0.719
-14.9	5.2	0.700
-13.8	7.2	0.735
-8.9	16.0	0.712
-8.0	17.6	0.735
-1.6	29.1	0.737
-0.4	31.3	0.704
4.8	40.6	0.729
5.9	42.6	0.715
11.1	52.0	0.736
12.1	53.8	0.732
17.6	63.7	0.719
18.6	65.5	0.716
23.9	75.0	0.729
24.9	76.8	0.706

Sample composition: $\text{CH}_3\text{N}_2\text{H}_3$ 84.7 w/o
 N_2H_4 14.0 w/o
 H_2O 1.0 w/o
 NH_3 0.1 w/o
Other soluble impurities 0.2± w/o
 (CH_3NH_2) Trace).

(U) The cell used in this program is shown schematically in Fig. 16. In this apparatus, the test fluid is contained in a thin annular passage between two aluminum alloy cylinders. The annulus is approximately 1 inch in diameter, 0.020 inch thick, and 5-3/4 inches long. The ends of the annulus are sealed with two Teflon O-rings, which hold the cylinders concentrically and minimize the heat conduction path between the cylinders. To maintain end effects at a minimum, two thermal barriers fabricated of Teflon are fitted over the ends of the cylinders. The cell is held together by two stainless-steel end plates which fit over the thermal barriers. Six pairs of copper-constantan thermocouples are embedded at various positions in both cylinders, close to the surface of the cavity containing the test fluid. Thermocouple wire diameter is maintained as small as possible to minimize heat losses. An electrical resistance heater, located in the inner cylinder, supplies the heat energy to establish a temperature gradient across the liquid layer. The temperature of the outer cylinder is regulated by immersion of the cell in a constant-temperature bath.

(U) The experimental procedure is straightforward but rather tedious. A sample of the test fluid is placed into a stainless-steel loading apparatus attached to the cell. By proper manipulation of the loading apparatus, the annulus is first evacuated, and then the test fluid is drawn into the cell. The cell is placed in the constant-temperature bath which is adjusted to some preselected and regulated temperature. Electrical power is applied to the cell heater through use of a regulated d-c power supply until a temperature gradient of the desired magnitude is obtained across the annulus. Temperature gradients are maintained at approximately 1 F to minimize convection. After thermal equilibrium is attained, measurements of heater voltage and current are made through use of a Leeds and Northrup K-3 potentiometer in conjunction with a precision volt box and current shunt. This instrument is also used to measure the temperature gradient across the annulus and the bath temperature.

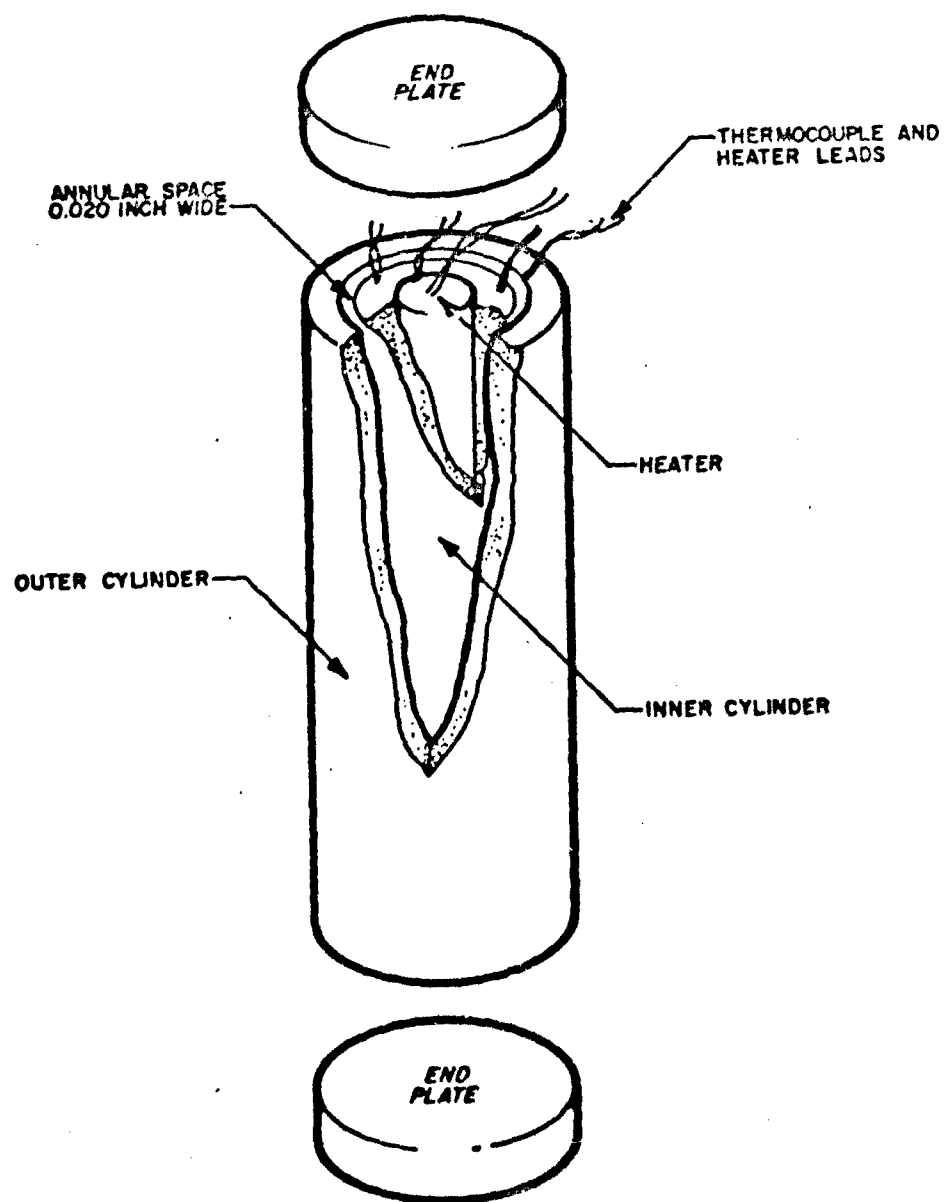


Figure 16. Thermal Conductivity Cell

- (U) The thermal conductivity of the test fluid is calculated through use of the equation:

$$k = \frac{Q \Delta X}{A \Delta T} \quad (21)$$

where

- k = thermal conductivity, Btu/hr-ft-F
Q = heat flux, Btu/hr
A = heat transfer area normal to heat flux, sq ft
 ΔX = liquid layer thickness, feet
 ΔT = temperature gradient, F

- (U) Prior to conducting actual thermal conductivity measurements on test fluids, a series of vacuum calibrations of the cell are made. These calibrations are necessary to account for cell heat losses along thermocouple wires, along heater lead wires, and through the ends of the cylinders. Calibrations are made at 50 F intervals throughout the temperature range of interest. Electrical power levels required to maintain given temperature gradients (~ 1 F) across the annulus are measured at each operating temperature. These heat losses are subtracted from the total heat input measured during actual thermal conductivity runs to obtain a net heat input.

- (U) Thermal Conductivity of UDMH. Thermal conductivity measurements were made on propellant-grade unsymmetrical dimethylhydrazine (UDMH), $(CH_3)_2N_2H_2$, over a nominal temperature range of 0.5 to 251.0 F; the results of these measurements are presented in Table 10. All UDMH samples were obtained from a single large quantity of propellant which was analyzed and found to be within specifications as presented in MIL-P-25604C. The analysis is presented in Table 10. The initial series of measurements were conducted on Sample A at 50 F intervals from 0 to 150 F. To increase the reliability of these data, measurements were attempted on another sample of propellant from the original propellant batch. However, after the

TABLE 10

EXPERIMENTAL THERMAL CONDUCTIVITY DATA

ON $(\text{CH}_3)_2\text{N}_2\text{H}_2$

Sample	Temperature, F	Thermal Conductivity, Btu/hr-ft-F
A*	0.6	0.101
A	0.6	0.107**
D*	0.5	0.100
D	0.5	0.100
A	51.1	0.095
A	51.1	0.098
B*	51.0	0.095
B	51.0	0.095
A	100.7	0.085
A	100.7	0.085
B	100.7	0.087
B	100.7	0.088
A	150.7	0.081
A	150.6	0.082
B	150.7	0.081
B	150.7	0.085
B	200.8	0.076
B	200.9	0.075
C*	200.9	0.073
C	200.9	0.072
B	251.0	0.068
B	251.0	0.066
C	251.0	0.066
C	251.0	0.066

*Sample Composition: $(\text{CH}_3)_2\text{N}_2\text{H}_2$ 99.8 w/oOther Soluble 0.2[±] w/o
Impurities $[\text{H}_2\text{O}]$ Trace] $[(\text{CH}_3)_2\text{NH}]$ Trace]

**Discarded data point; explanation contained in text

sample was placed in the cell and measurements resumed, abnormal thermocouple output voltage signals were obtained.

- (U) A search for the cause of the difficulty was initiated and centered about the electrical components (and electrical connectors) associated with the cell. After this search revealed no apparent problem areas, the problem was traced to incomplete filling of the cell annulus with the liquid propellant. As a result, some thermocouples were reading the temperature drop across a liquid layer while others were reading the temperature drop across a vapor layer. The cause of the difficulty occurred during propellant loading operations, when a valve, located between the cell annular gap and vacuum pump, apparently leaked, and a portion of the propellant sample was drawn out of the cell. The malfunction was corrected, and measurements were resumed on a new propellant charge (Sample B).
- (U) Thermal conductivity measurements were made using Sample B at temperatures of approximately 50, 100, 150, 200, and 250 F. The data obtained with samples A and B compared favorably in the overlapping range, i.e., from 50 to 150 F. A third propellant charge, Sample C, of the original batch was placed in the cell, and measurements were made at 200 and 250 F. Again, the data compared favorably with the data obtained with Sample B at 200 and 250 F. Sample D was placed in the cell and duplicate measurements at the 0 F temperature level were made. One data point taken at this temperature level was discarded because of its disagreement with the other data and because its deviation from the average was greater than twice the standard deviation.
- (U) The valid data were curve fit from 0.5 to 251.0 F, and a graphical presentation is shown in Fig. 17. The equation which represents the data is:

$$k(\text{Btu/hr-ft-F}) = 0.1014 - 1.368 \times 10^{-4} t(\text{F}) \quad (22)$$

The standard deviation of the least squares curve fit is 0.0017 Btu/hr-ft-F.

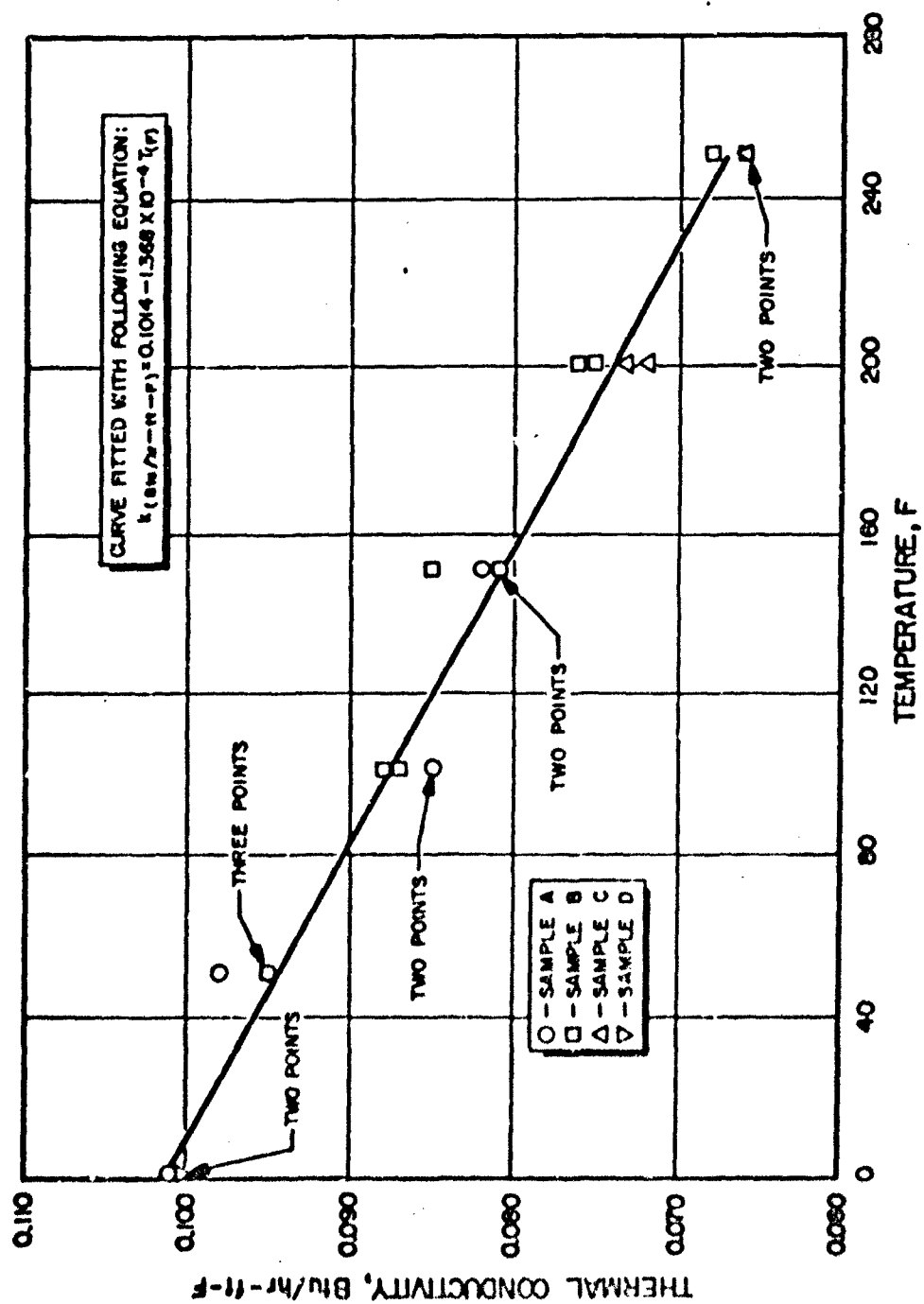


Figure 17. Thermal Conductivity of $(CH_3)_2N_2H_2$

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- (C) Thermal Conductivity of MIF-3. Following completion of measurements on UDMH, thermal conductivity determinations were made on the MIF-3 fuel blend (nominally 86 w/o MMH-14 w/o hydrazine). Measurements were conducted on two different propellant-grade samples (A and B) at approximately 50 F intervals over the temperature range of 0.4 to 251.3 F. The results of these measurements, presented in Table 11, show that the thermal conductivity of MIF-3 ranges from 0.170 to 0.128 Btu/hr-ft-F over this temperature range.

- (C) The data were curve fit from 0.4 to 251.3 F and a graphical presentation of the data is shown in Fig. 18. The equation which represents the data is:

$$k(\text{Btu/hr-ft-F}) = 0.1688 - 1.063 \times 10^{-4} t_{(F)} - 1.891 \times 10^{-7} t_{(F)}^2 \quad (23)$$

The standard deviation of the least squares curve fit is 0.0025 Btu/hr-ft-F.

- (U) During the measurements on propellant sample B, unstable thermocouple voltages were observed. This was finally traced to a poor electrical connection in the apparatus measurement system. After repair and checkout tests (in which the apparatus functioned normally), the MIF-3 thermal conductivity measurements were completed.
- (C) The two MIF-3 samples used in the measurements were obtained from a specific batch of propellant prepared for this purpose. The MIF-3 fuel blend, which has a nominal composition of 86 w/o monomethylhydrazine, $\text{CH}_3\text{N}_2\text{H}_3$, and 14 w/o hydrazine, N_2H_4 , was blended from propellant grade samples of $\text{CH}_3\text{N}_2\text{H}_3$ and N_2H_4 . The blend was prepared under a nitrogen atmosphere in a dry box to prevent moisture absorption and fuel reaction with oxygen and carbon dioxide in the air. The results from chemical analysis of the homogeneous blend are listed in Table 11.
- (C) Thermal Conductivity of MIF-5. Subsequent to the completion of measurements on MIF-3, thermal conductivity determinations were initiated on the MIF-5 fuel blend using the same cell and experimental technique. Because a sample of the MIF-5 fuel, which has a nominal composition of 55 w/o $\text{CH}_3\text{N}_2\text{H}_3$, 26 w/o

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TABLE 11

EXPERIMENTAL THERMAL CONDUCTIVITY DATA ON MHF-3

Sample	Temperature, F	Thermal Conductivity, Btu/hr-ft-F
A*	0.4	0.166
A	0.4	0.166
B*	0.7	0.167
B	0.7	0.170
A	51.0	0.166
A	50.9	0.163
B	51.1	0.168
B	51.1	0.166
A	100.7	0.154
A	100.7	0.156
B	100.6	0.155
B	100.6	0.160
A	150.7	0.146
A	150.7	0.148
B	150.6	0.146
B	150.6	0.146
A	201.1	0.141
A	201.1	0.142
B	201.2	0.138
B	201.2	0.136
A	251.2	0.134
A	251.2	0.132
B	251.3	0.128
B	251.3	0.130

*Sample composition:

$\text{CH}_3\text{N}_2\text{H}_3$	85.1 w/o
N_2H_4	13.9 w/o
H_2O	0.8 w/o
NH_3	0.1 w/o
Other Soluble Impurities	0.1 w/o
(CH_3NH_2)	Trace)

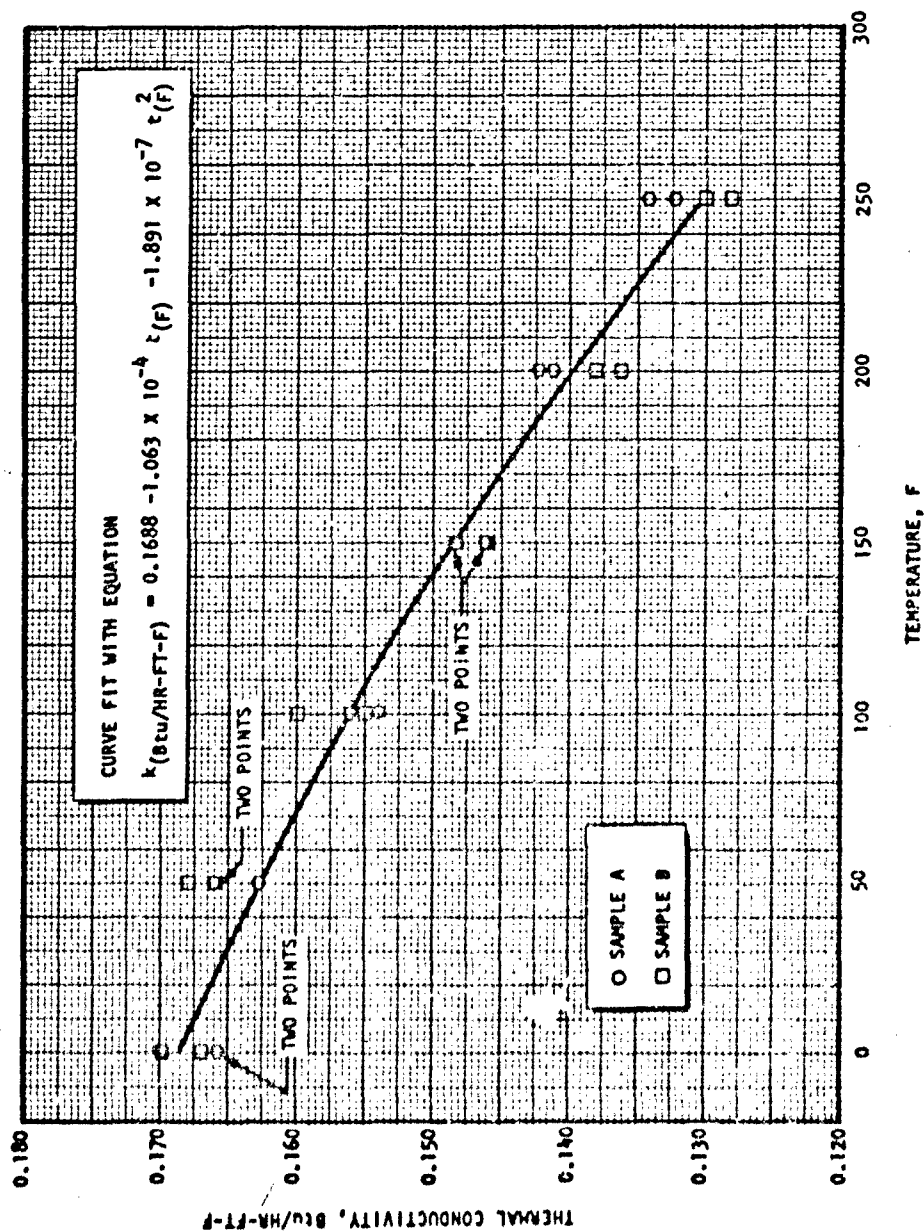


Figure 18. Thermal Conductivity of MUF-3

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N_2H_4 , and 19 w/o $N_2H_4 \cdot HNO_3$, was not available, a small sample was prepared from propellant-grade samples of $CH_3N_2H_3$ and N_2H_4 , and an analytical grade sample of NH_4NO_3 . The sample was prepared in an inert atmosphere of dry nitrogen in a dry box to prevent the propellants from absorbing moisture and from reacting with oxygen and carbon dioxide in the air. Because $N_2H_4 \cdot HNO_3$ was not available, NH_4NO_3 was used to prepare the hydrazine salt. The NH_4NO_3 was added to the $CH_3N_2H_3$, rather than to the N_2H_4 , to minimize the salt concentration and hence the impact sensitivity hazard. The temperature of the solution was continuously monitored during the addition of NH_4NO_3 and a slight temperature rise was noted. Ammonia produced in the solution was reduced to an acceptable level by vacuum distillation. The blending process was completed by adding $CH_3N_2H_3$ (lost during the distillation) and N_2H_4 . The chemical analysis of the resulting fuel blend is presented in Table 12.

- (C) An initial series of measurements was conducted on a sample (designated as Sample A) of MIF-5 at approximately 50 F intervals over a temperature range extending from 0.5 to 201.0 F; the results of these measurements are presented in Table 12. Preliminary test results indicate that the thermal conductivity of MIF-5 varies from 0.188 to 0.171 Btu/hr-ft-F over this range. To increase the reliability of these data, another series of measurements will be made over approximately the same temperature range on a second sample of propellant from the same batch of the MIF-5 fuel blend. The thermal conductivity cell was thoroughly flushed and dried in preparation for this second series of measurements. Following loading of the cell with propellant, thermal conductivity measurements will be continued on MIF-5.

(U) Viscosity

Under the present program, viscosity measurements have been planned for a variety of propellants at extended temperatures and pressures. To satisfy all potential viscosity requirements, an all-metal capillary viscometer was designed for the measurements. The capillary flow technique was selected because of the relative simplicity of the viscometer's construction and operation.

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TABLE 12

PRELIMINARY EXPERIMENTAL THERMAL CONDUCTIVITY DATA ON MHF-5*

Sample	Temperature, F	Thermal Conductivity, Btu/hr-ft-F
A*	0.5	0.187
A	0.5	0.188
A	50.9	0.185
A	50.8	0.185
A	100.6	0.180
A	100.6	0.180
A	150.5	0.175
A	150.5	0.176
A	200.8	0.172
A	201.0	0.171

*Sample composition:

$\text{CH}_3\text{N}_2\text{H}_3$	54.9 w/o
N_2H_4	25.4 w/o
$\text{N}_2\text{H}_4 \cdot \text{HNO}_3$	18.9 w/o
NH_3	0.2 w/o
H_2O	0.6 w/o

- (U) The design of the apparatus is shown schematically in Fig. 19. In this apparatus, the viscosity is obtained by observing the flowrate through the capillary tubing and the corresponding driving fluid head, which in this apparatus, is a simple gravity head resulting from a difference in the elevation of the liquid level in the two legs of the U-tube. The reservoir in one of these legs is a section of 0.75-inch tubing which contains a magnetic float at the gas-liquid interface. The position of this float within a vertical range of approximately 6 inches is sensed by a differential transformer unit surrounding the tubing. For the other leg of the U-tube, valves B and C provide a choice between a 1-1/2-inch and 3/8-inch tubing reservoir.

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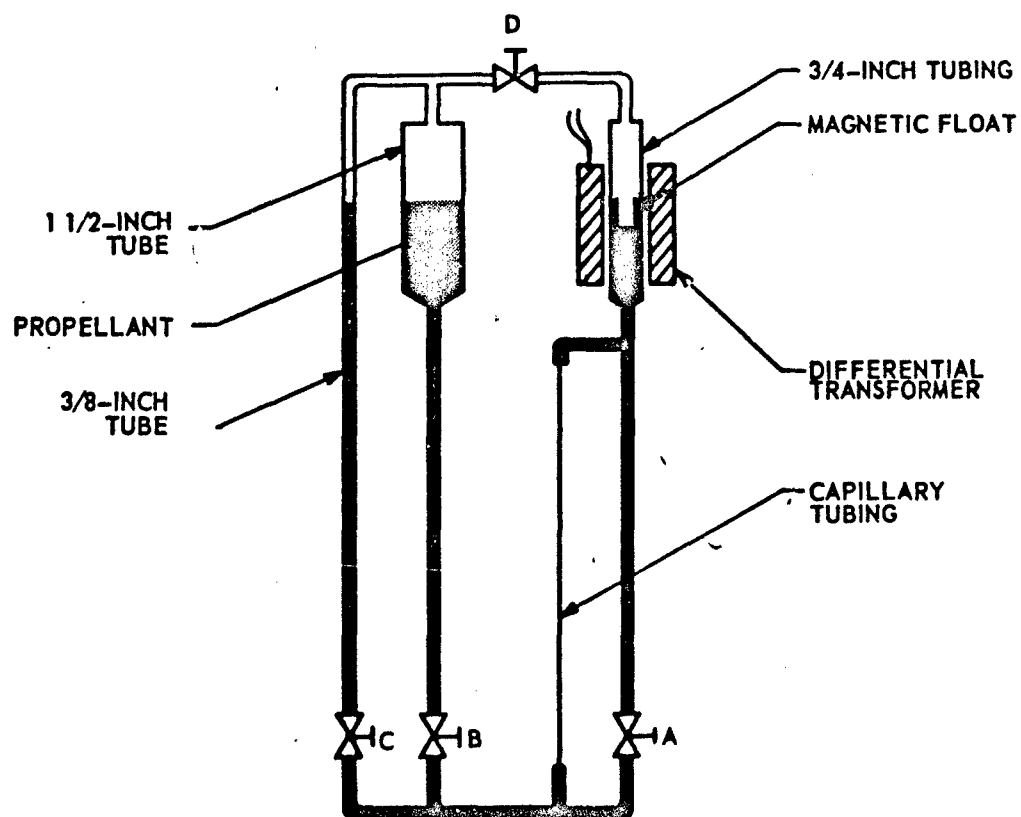


Figure 19. Capillary Viscometer Schematic

- (U) The viscometer is constructed entirely of stainless-steel tubing and fittings to permit testing corrosive liquids at pressures up to 1000 psi. The capillary tubing is 0.023-inch ID and approximately 26-1/2 inches long. This long, large-bore capillary was selected in an attempt to minimize the relative importance of entrance and exit friction losses at low kinematic viscosities, which may approach 0.1 centistoke for propellants of interest. For the case of flow between the 3/4-inch float reservoir and the 1-1/2-inch reservoir, this capillary would lead to experimental flow times ranging from minutes to hours in the viscosity range contemplated. To increase the speed of the test process at the higher viscosities, the option of the 3/8-inch reservoir has been provided, which permits setting up a much larger differential head within the operating range of the transducer and float. In addition, this option permits testing of a given viscosity under significantly different driving heads, flow velocities, and hence, Reynolds numbers, for the capillary. This feature could permit estimation or calibration of the end effects at low viscosities. It may be possible to extend the applicability of the viscometer into a range where end effects are important through a technique of self-calibration, in which a given viscosity is measured over a sufficiently wide Reynolds number range.
- (U) Not shown in the figure are accessory connections and valving for loading, venting, and/or pressurizing the viscometer with inert gas. The overall unit, approximately 5 feet tall, is housed in a temperature-controlled dry box equipped with heater, circulation fans, thermocouples, pressure transducer, etc.
- (U) In operation, the viscometer is filled with liquid propellant to an elevation within the sensing range of the float and transformer. A differential head is established between the float reservoir and one of the other reservoirs by inert gas pressurization or by venting propellant vapors from one of the reservoirs. Subsequently, this head is allowed to dissipate by flow through the capillary tubing. A line and valve (D) connecting the gas spaces of the float reservoir and the other reservoirs separate these spaces while establishing the initial liquid head, and permit equalization of the pressure over the liquid during the capillary-flow process. The

flow through the capillary may be in either direction; the choice of flow direction determines the elevation of initial fill, or equilibrium liquid level position, within the range of the float and transformer.

- (U) Calibration of the relative volumetric capacities of the U-tube legs establishes differential liquid heads as a function of float position with reference to the equilibrium position. Valve A allows bypassing the capillary tubing to facilitate the processes of establishing a differential head and the equilibrium position. Viscosity is determined from the time history of head dissipation in flow through the capillary plus calibration of its flow resistance.
- (U) The flow process leads nominally to an exponential decay of liquid head with time which may be given as

$$\frac{d}{dt} (\ln x) = -\frac{C}{\nu} \quad (24)$$

where x is float displacement from the equilibrium position, t is time, ν is the kinematic viscosity of the liquid, and C is a nominally constant factor involving fundamental constants and instrument parameters (capillary length and radius, and areas of the U-tube reservoir arms).

- (U) The principal calibration factor, C , was obtained from tests with a liquid of known kinematic viscosity. Calibration runs were made with deionized water at temperatures of approximately 65 F (kinematic viscosity approximately 1.0 centistoke), 110 F (0.6 centistoke), and 160 F (0.4 centistoke). Several tests were conducted at each viscosity level using each of the optional secondary reservoirs which complete the U-tube with the float reservoir.
- (U) The performance of the viscometer was generally satisfactory during these tests; the exponential decay of float position with time was accurately realized; no float sinkings were experienced. Values derived for the principal calibration factors (C corresponding to each optional secondary arm) were consistent within 3 percent, and agree reasonably well with

estimates based on dimensions of the apparatus. The scatter in experimental values was largely due to uncertainty or variation in the temperature, and hence the viscosity, for a run. Temperature has negligible direct effect on the instrument parameters.

- (U) Viscosity of Chlorine Pentafluoride. After apparatus calibrations had been completed, viscosity measurements were initiated on chlorine pentafluoride, ClF_5 , to extend the experimental data above the presently available temperature range, -130.5 to 68°F (Ref. 1). During initial determinations with ClF_5 the instability of the open-cup float was found to be a serious experimental problem. It was assumed that the float would sink if sufficient liquid were allowed to condense inside it, and this condition would be more likely to occur with relatively high vapor pressures (e.g., at higher temperatures and with relatively volatile liquids like ClF_5). Thus, precautions were observed in the experimental procedures to prevent this occurrence. However, despite these preventative measures, the float consistently failed to remain afloat during the first trial runs with ClF_5 . Upon disassembly of the apparatus it was found that float sinking was actually due to leakage of liquid through a corrosion pinhole. After replacing the float, sinkings have only been experienced during operations involving thermal fluctuations, as in heating the apparatus and setting up the initial liquid head. More efficient experimental procedures are being developed to overcome these difficulties.
- (U) In the initial viscosity determinations on ClF_5 , it was observed that the viscometer can apparently operate near 0.1 centistoke without introducing significant end-effect problems. During tests near this level, a substantial portion of the float travel follows the exponential decay law expected with simple streamline flow resistance. As a result of these determinations, two viscosity points have been obtained for propellant-grade ClF_5 at 154 and 176°F under saturated liquid conditions. These data are presented in Table 13.
- (U) Each data point in Table 13 represents three measurements within $\pm 1^\circ\text{F}$ and better than 1 percent precision. Density data used for the reduction

of kinematic viscosity to absolute viscosity was taken from Ref. 8. The correlation of these initial data with the previously reported data (Ref. 1) appears reasonable; however, additional measurements must be obtained in the present viscometer to provide a reliable extension of the ClF_5 viscosity data over the complete temperature range.

TABLE 13

EXPERIMENTAL CHLORINE PENTAFLUORIDE VISCOSITY DATA

Temperature, F	Kinematic Viscosity, centistokes	Absolute Viscosity, centipoises
154	0.134	0.215
176	0.121	0.185

PHASE III: EVALUATION AND COMPILATION OF DATA

OBJECTIVE

- (U) During the entire period of the contractual program, efforts under Phase III have been directed toward the assembly of all data generated during Phases I and II, verification of all the data sources, critical evaluation and comparison of conflicting data, and tabulation and correlation of the results. Where final selection of the best values has been completed, they are presented in this report, as discussed in the following paragraphs.

RESULTS AND ACCOMPLISHMENTS

- (U) Concurrently with the data evaluation of Phase III, comprehensive bibliographies of physical properties data are being compiled from references obtained through the Phase I literature survey. Each selected propellant is being described with a list of references for each of its individual physical properties. These compilations are not only useful to those who use the data, but also provide guides for the final assembly of data.
- (U) As these bibliographies are compiled, all of the original sources of referenced data are ordered, if they are not already on file, and checked for accuracy and originality of data. Secondary sources are used only where primary sources are not available and are referenced as such. As far as can be determined from a preliminary evaluation of the data, completely unreliable data are not used.
- (U) Using these bibliographies as guides, the physical properties are then compiled into data sheets. All of the available data are critically evaluated and presented in both metric and English units. When agreement among various sets of data is good, these data are combined and curve fit with a least-squares curve fit computer program. The resulting equations are included with the graphical representations of the data.

- (U) During the initial term of this contract, physical properties bibliographies were prepared for B_2H_6 (Appendix A) and N_2H_4 (Appendix B), and data sheets were compiled for N_2H_4 (Appendix C). In addition, preparation of a bibliography on N_2O_4 physical properties has been initiated.

Diborane

- (U) Except for the references cited under "General Reviews, Bibliographies, and Physical Property Compendia," and some of the references appearing under "General Chemistry, Reactions and Theory," the referenced data in the B_2H_6 bibliography (Appendix A) are original. All secondary sources are noted as such.
- (U) It should be noted that references to B_2H_6 heat of formation are limited to two primary sources and a compendium. The JANAF Thermochemical Data tables, which are accepted as the recommended source for thermochemical data, include a weighted value from the references presented in the bibliography and other primary sources. The two primary sources selected for inclusion in the bibliography represent the experimental values of direct determinations.

Hydrazine

- (U) In the N_2H_4 bibliography (Appendix B), the work of Audrieth and Ogg has been referenced several times as a secondary source of data. In these instances, extensive data are presented along with curve-fit equations for the particular property. It should also be noted that there is a disagreement among the values for the heat of fusion of N_2H_4 . Scott and co-workers obtained a value of 3025 cal/mole for the heat of fusion of N_2H_4 at the melting point. Previously, values of 1000 and 1020 cal/mole had been reported by Giguere, and Hieber and Woerner, respectively.
- (U) Using the bibliography as a guide, a compilation of N_2H_4 physical properties data was prepared. These data sheets (Appendix C) presented in both metric and English units, are Rocketdyne's recommendations for the best-value data

for the physical properties of hydrazine. All available properties data for hydrazine were critically evaluated, and the most representative values are included in the data sheets.

Nitrogen Tetroxide

- (U) Compilation of a physical properties bibliography for nitrogen tetroxide (N_2O_4) has been initiated. All of the references obtained through the literature survey are being placed on indexed punch cards.

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APPENDIX A

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APPENDIX B

N_2H_4 PHYSICAL PROPERTY BIBLIOGRAPHY

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Molecular Weight

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APPENDIX C

PHYSICAL PROPERTIES OF HYDRAZINE

- (U) The physical properties of hydrazine are presented in Table C-1. Figures C-1 through C-8a are graphical illustrations of properties listed in Table C-1.

TABLE C-1

PHYSICAL PROPERTIES OF HYDRAZINE

Property	Value		Figure Number	Reference Number
	Metric	English		
<u>General Identification</u>				
Identification	Hydrazine (H ₂)			
Molecular Formula	N ₂ H ₄			
Molecular Weight	32.046			
Freezing (Melting) Point	1.53 C	34.8 F		C-1
Normal Boiling Point	112.6 C*	234.7 F*		C-2
Triple Point				C-2, -3, -4
Critical Point				
Temperature	300 C	716 F		C-5
Pressure	105 atm	2130 psia		C-6
Density	0.231 gm/cc	16.42 lb/cu ft		C-7
<u>Phase Properties</u>				
Density				
Solid	1.166 gm/cc at -5 C	71.54 lb/cu ft at 23 F		C-8
Liquid	1.0046 gm/cc at 25 C*	62.645 lb/cu ft at 77 F*	C-1, -1a	C-9, -10, -11
Vapor (90 to 130 C)	$P(\text{gm l}) = 0.08305 \times T(\text{K})$	$P(\text{lb/cu ft}) = \frac{32.046 \times P(\text{psia})}{10.73 \times T(\text{R})}$		C-12
Vapor Pressure				
Solid	2.59 mm Hg at 0 C	0.050 psia at 32 F		C-2
Liquid	16.00 mm Hg at 25 C*	0.271 psia at 77 F*	C-2, -2a	C-2, -3, -4
Surface Tension	66.67 dyne/cm at 25 C	0.005568 lb/ft at 77 F		C-13
	62.32 dyne/cm at 35 C	0.005270 lb/ft at 95 F		C-14
	$9.536 \times 10^{-5} \text{ atm}^{-1}$ at 25 C	$5.30 \times 10^{-4} \text{ psi}^{-1}$ at 77 F		C-15
<u>Coefficient of Thermal Expansion (90.5 percent N₂H₄)</u>				
Compressibility				
Adiabatic	$2.309 \times 10^{-5} \text{ atm}^{-1}$ at 25 C	$0.1572 \times 10^{-5} \text{ psi}^{-1}$ at 77 F		C-16
Isothermal	$2.542 \times 10^{-5} \text{ atm}^{-1}$ at 25 C	$0.1730 \times 10^{-5} \text{ psi}^{-1}$ at 77 F		C-16

NOTE: Data in this table marked with an asterisk are based on Benedictyne interpolation of referenced data.

TABLE C-1
(Continued)

Property	Value		Figure Number	Reference Number
	Metric	English		
<u>Thermodynamic Properties</u>				
<u>Heats of Formation</u>				
Liquid	12.05 kcal/mole at 25 C	676.79 Btu/lb at 77 F		C-17
Gas	22.750 kcal/mole at 25 C	1277.7 Btu/lb at 77 F		C-2
Fusion	3025 cal/mole at melting point	169.9 Btu/lb at melting point		C-2
Sublimation				
Vaporization	10.7 kcal/mole at 25 C	601.0 Btu/lb at 77 F		C-2
Combustion	-186.635 kcal/mole at 25 C	-9348.2 Btu/lb at 77 F		C-18
<u>Heat Capacity</u>				
Solid	12.195 cal/mole-C at 200 K	0.381 Btu/lb-F at 99.4 F	C-3	C-2
Liquid	0.7563 cal gm-C at 25 C	0.7563 Btu/lb-F at 77 F	C-4, -4a	C-2, -19
Gas-Cp	12.6 cal/mole-C at 25 C	0.393 Btu/lb-F at 77 F	C-5, -5a	C-2
Cv	9.6 cal/mole-C at 12 C	0.300 Btu/lb-F at 107.6 F		C-20
Enthalpy (Gas)	---	---	C-6	C-2
Entropy				
Liquid	28.97 cal/mole-C at 25 C	0.904 Btu/lb-F at 77 F		C-2
Gas	56.97 cal/mole-C at 25 C	1.778 Btu/lb-F at 77 F		C-2
<u>Entropy of Fusion</u>				
	11.01 cal/mole-C at melting point	0.3435 Btu/lb-F at melting point		C-2
Vaporization	35.89 cal/mole-C at 25 C	1.120 Btu/lb-F at 77 F		C-2
<u>Transport Properties</u>				
<u>Viscosity</u>				
Liquid	0.870 centipoise at 25 C	5.85×10^{-4} lb/ft-sec at 77 F	C-7, -7a	C-9, -21
Gas	---	---		
<u>Thermal Conductivity</u>				
Liquid	1.170×10^{-3} cal/sec-cm-C at 25 C	0.283 Btu/hr-ft-F at 77 F	C-8, -8a	C-22
Gas	3.49×10^{-5} cal/sec-cm-C at 25 C	8.44×10^{-5} Btu/hr-ft-F at 77 F		C-23

TABLE C-1
(Concluded)

Property	Value		Figure Number	Reference Number
	Metric	English		
Velocity of Sound (Liquid)	2090 m/sec at 25 C	6857 ft/sec at 77 F		C-16
Electromagnetic Properties				
Index of Refraction	$n_D = 1.4676$ at 25 C (77 F)*			C-18, C-24
Dipole Moment	1.90 ± 0.05 Debye			C-25
Dielectric Constant	31.7 at 25 C (77 F)			C-26
Electrical Conductivity	$1.5-3.0 \times 10^{-6} \text{ ohm}^{-1} \text{ cm}^{-1}$ at 25 C (77 F)			C-11, C-27
Magnetic Susceptibility				

Chemical Analysis

Appearance: Clear, oily, water-white liquid
 Odor: Similar to ammonia
 Composition: Military Specification (Ref. A-28)
 97.5 w/o minimum hydrazine
 2.5 w/o maximum water and other soluble impurities
 1.004 ± 0.002 gm/cc density at 25 C (77 F)

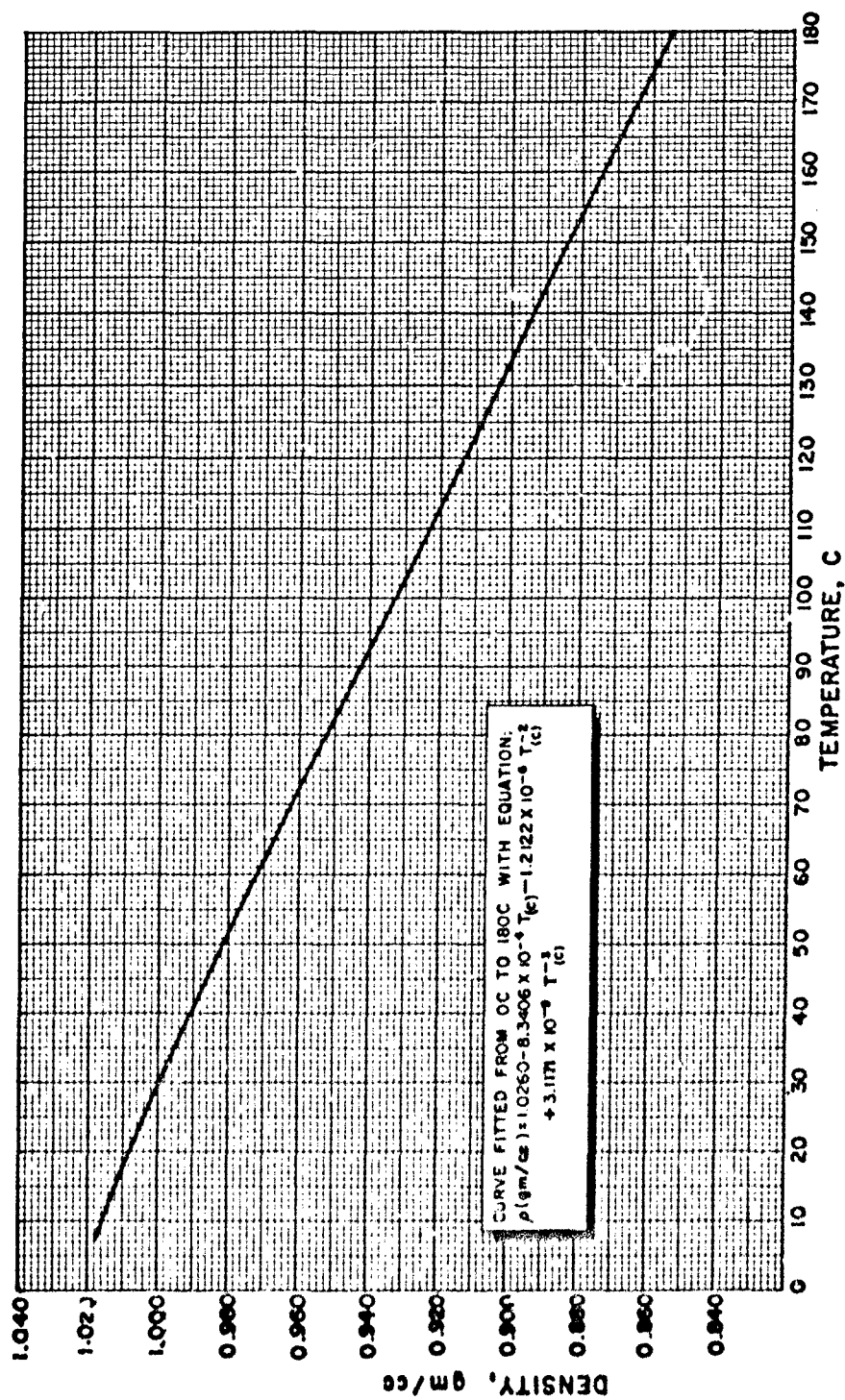


Figure C-1. Density of Anhydrous Hydrazine (Ref. C-9 through C-11)

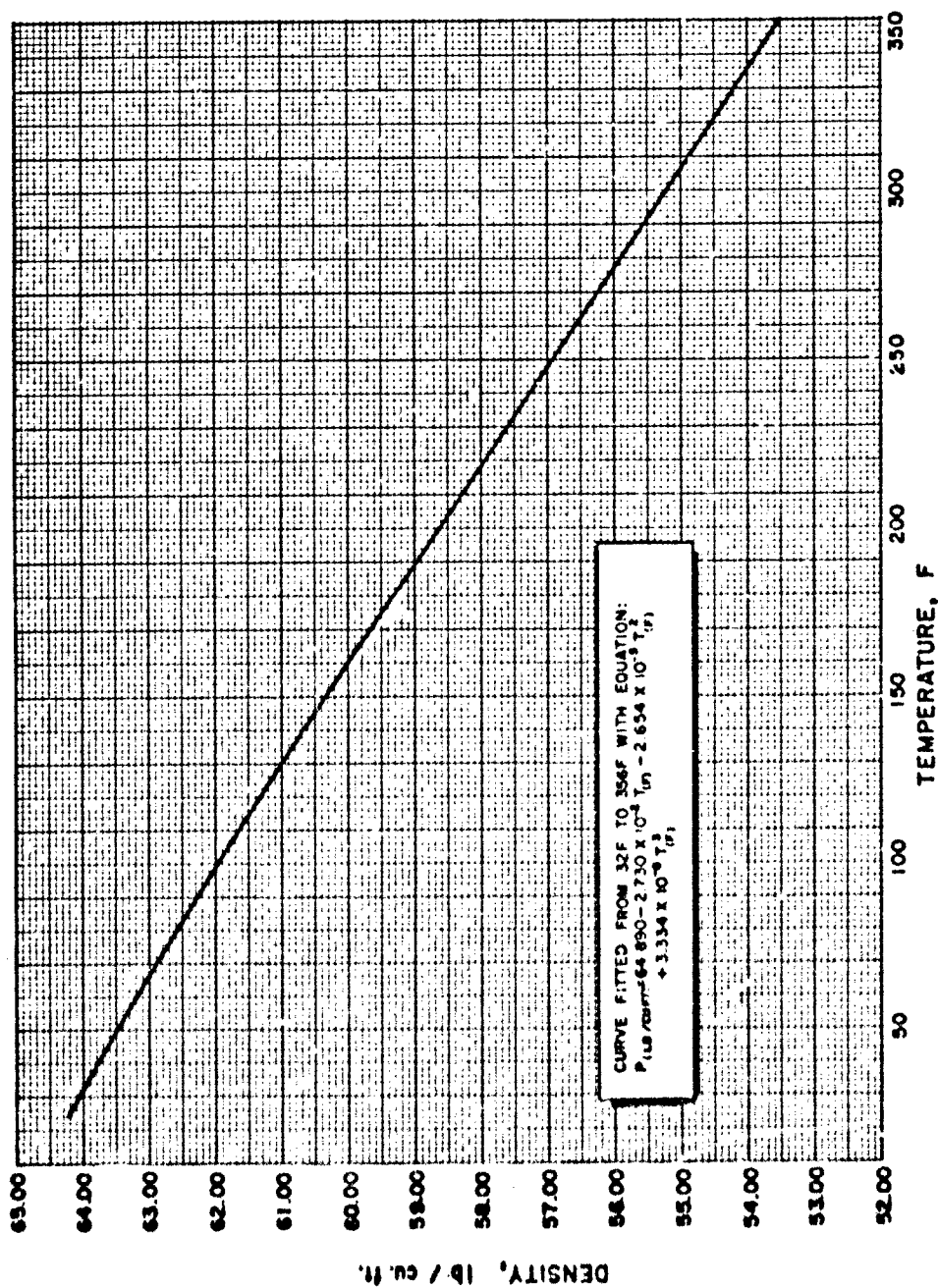


Figure C-la. Density of Anhydrous Hydrazine (Ref. C-9 through C-11)

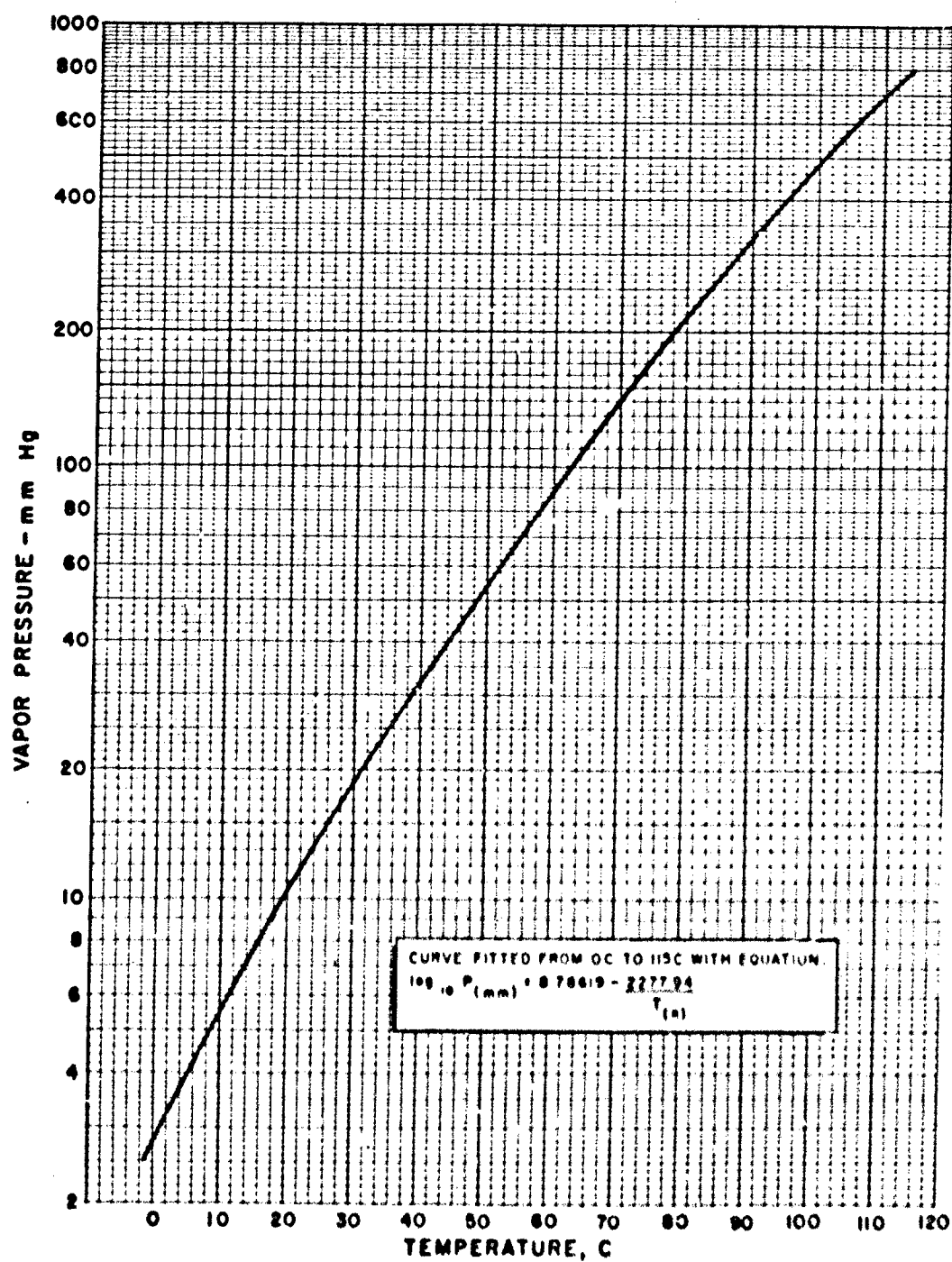


Figure C-2. Vapor Pressure of Anhydrous hydrazine (Ref. C-2 through C-4)

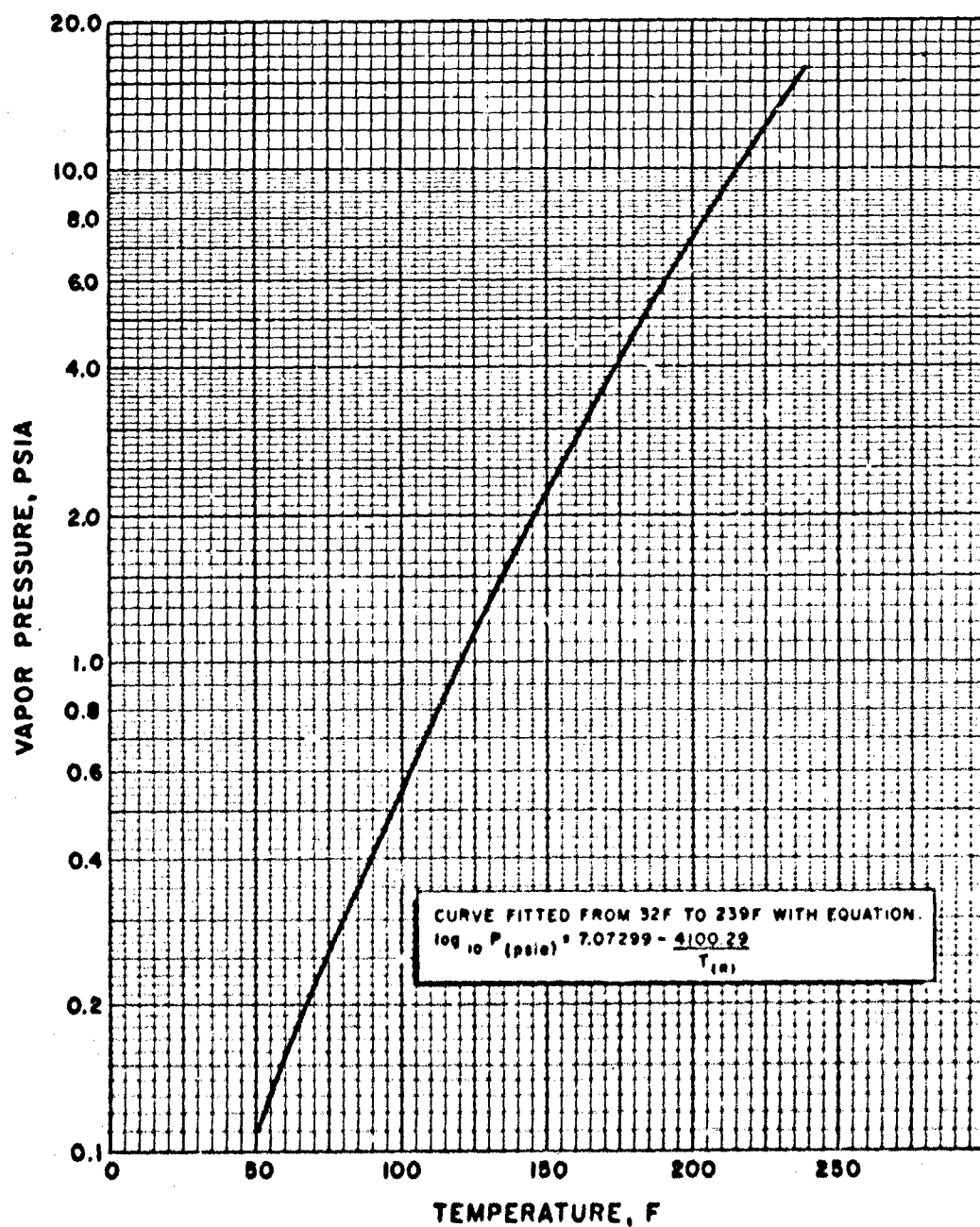


Figure C-2a. Vapor Pressure of Anhydrous Hydrazine (Ref. C-2 through C-4)

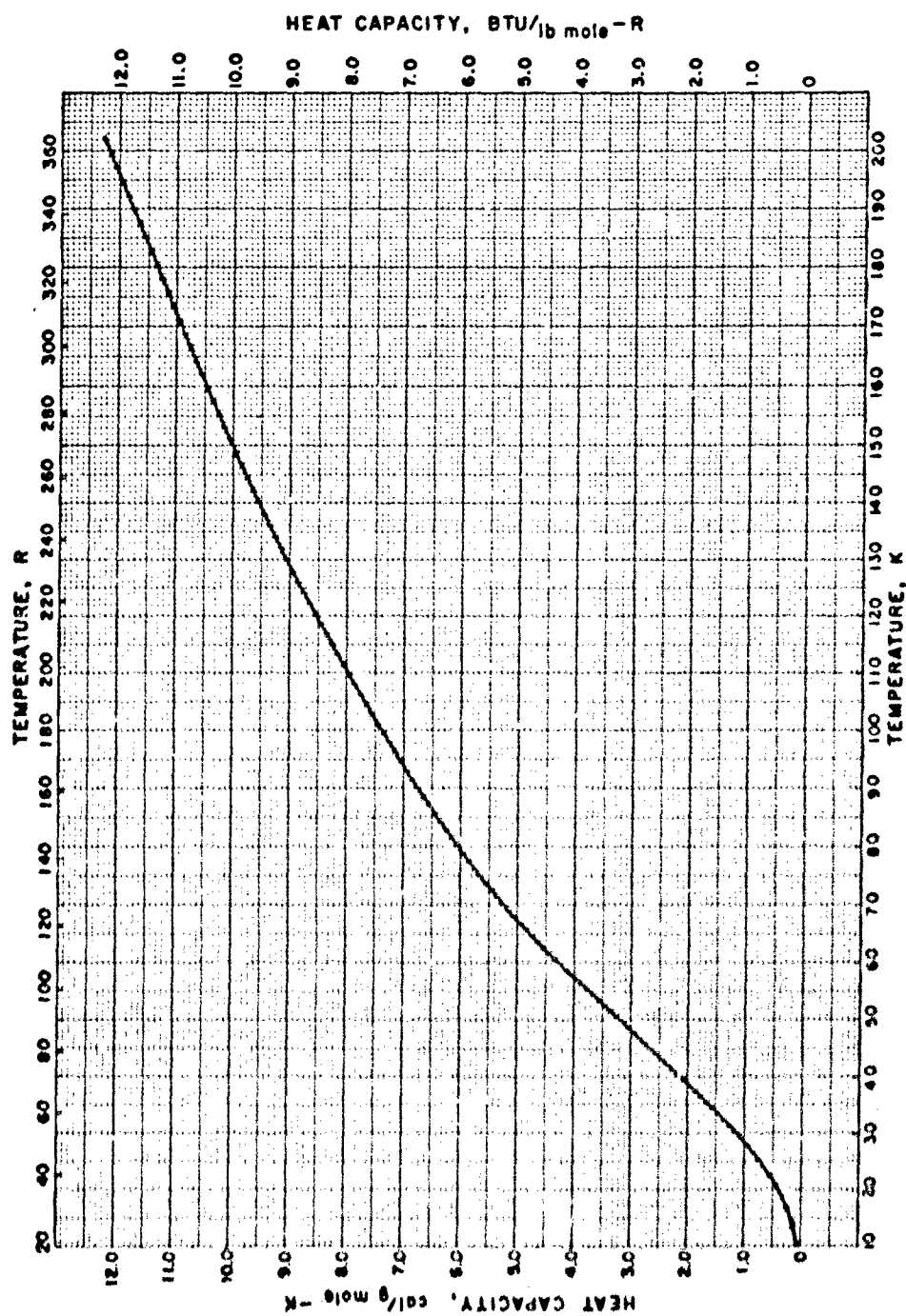


Figure C-3. Heat Capacity of Anhydrous Hydrazine--Solid Phase--(Ref. C-2)

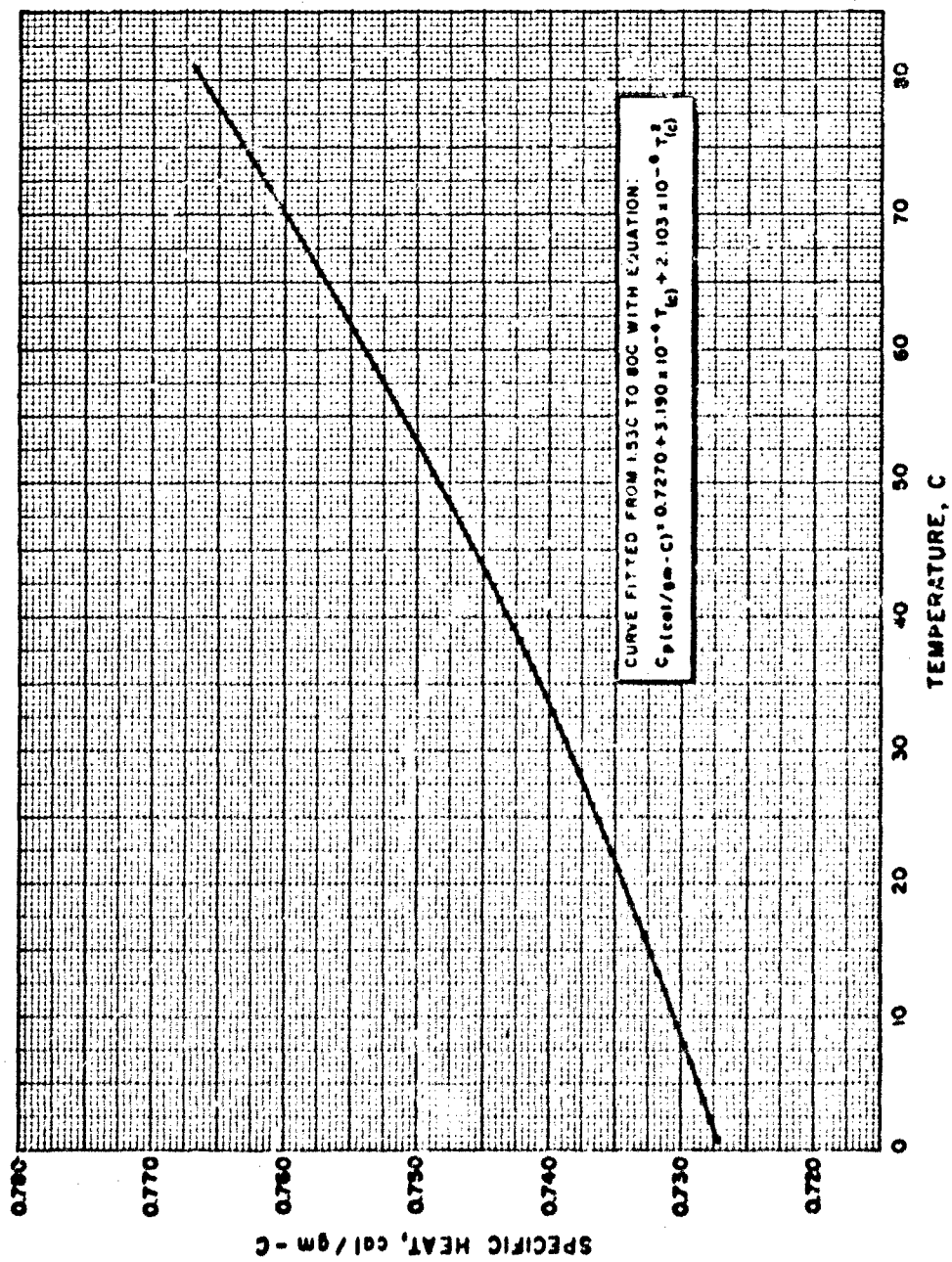


Figure C-4. Specific Heat of Anhydrous Hydrazine—Liquid Phase—(Ref. C-2 and C-19)

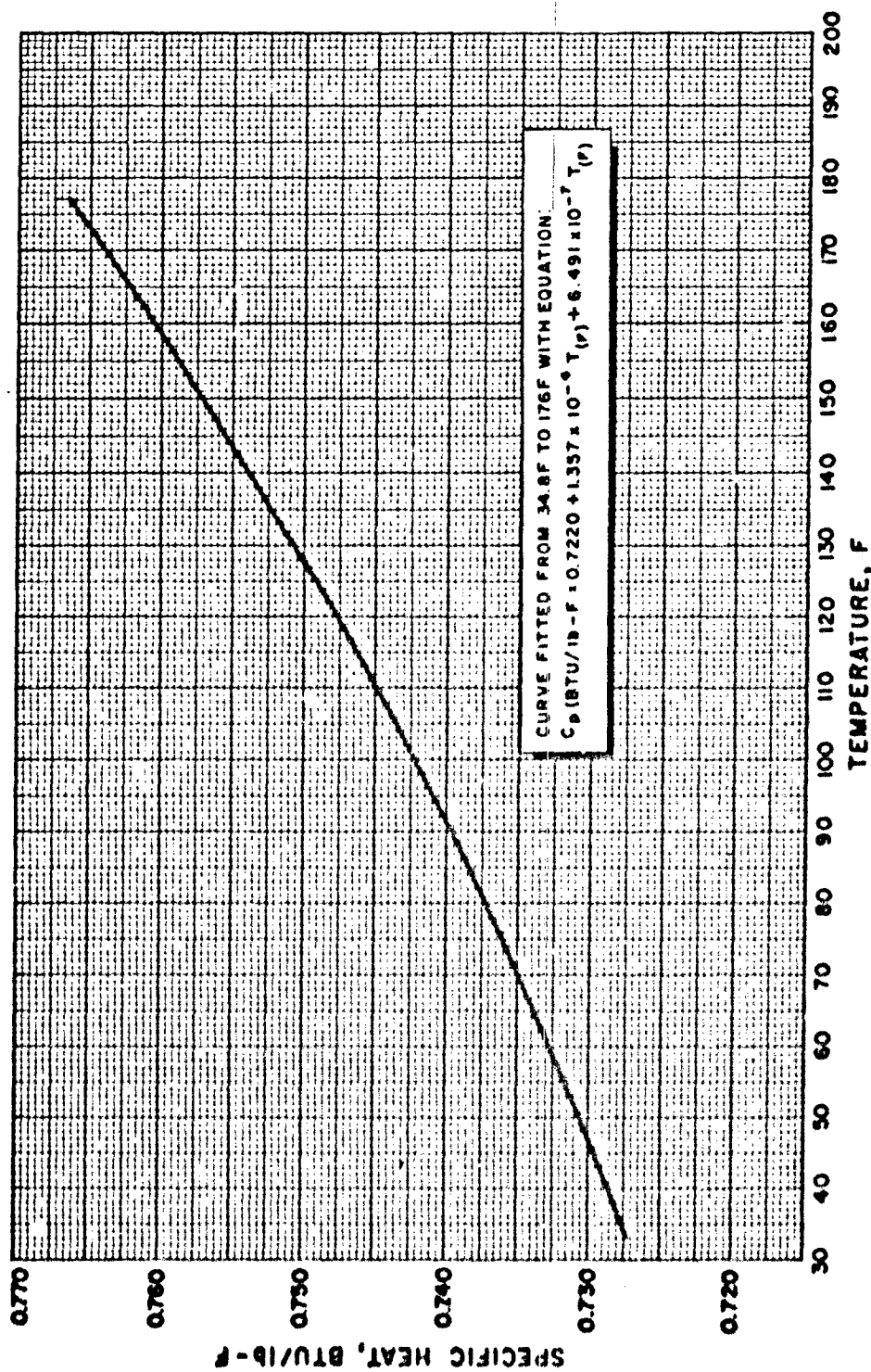


Figure C-4a. Specific Heat of Anhydrous Hydrazine--Liquid Phase--
 (Ref. C-2 and C-19)

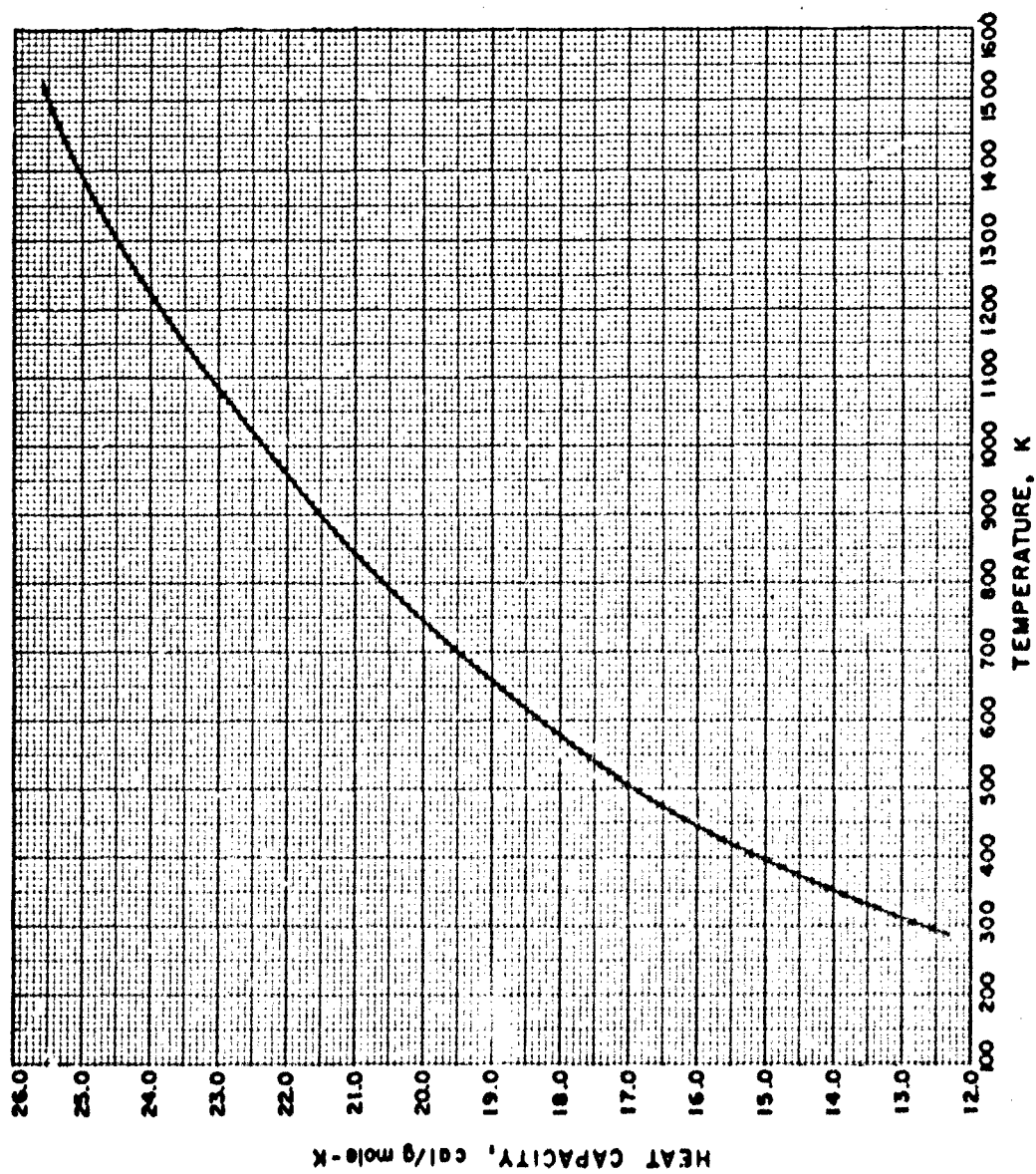


Figure C-5. Heat Capacity of Anhydrous Hydrazine--Vapor Phase--(Ref. C-2)

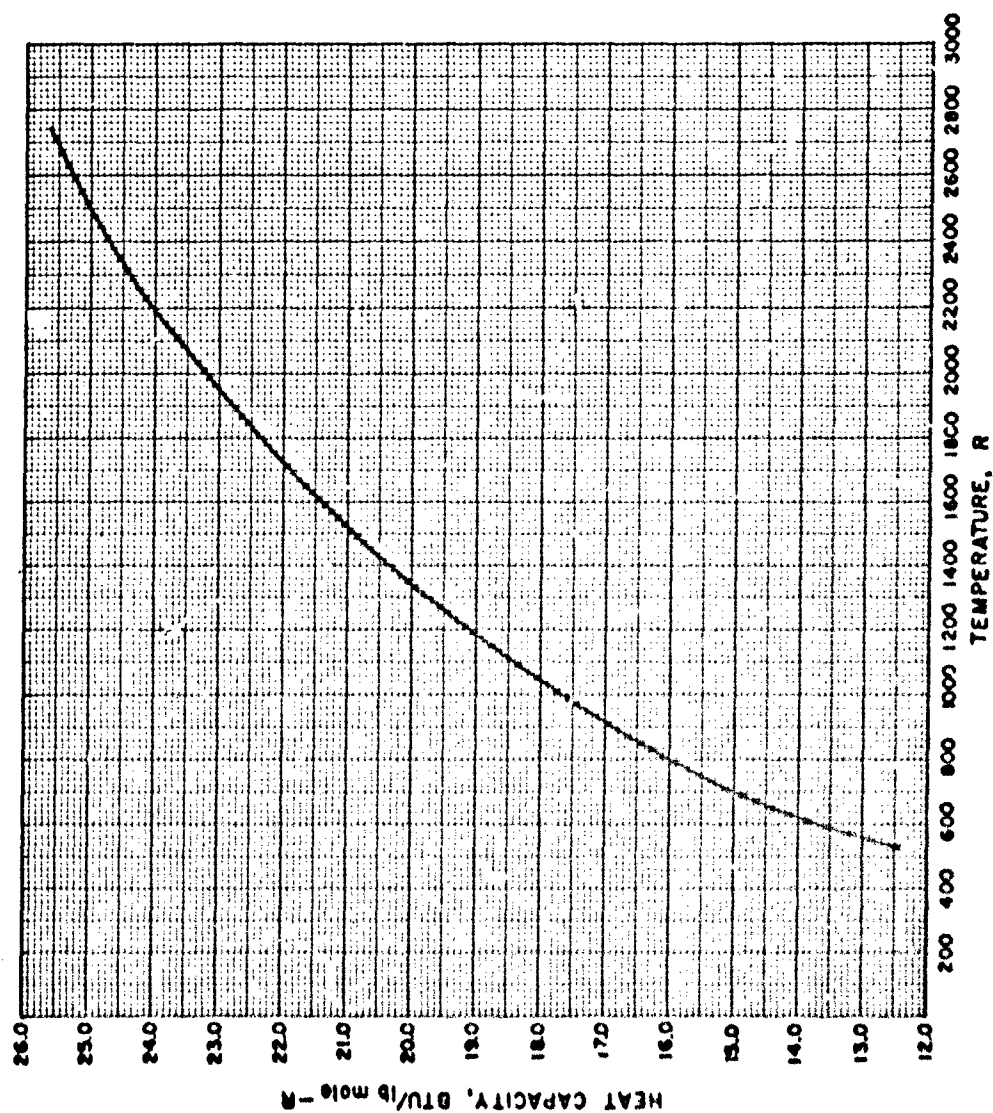


Figure C-5a. Heat Capacity of Anhydrous Hydrazine--Vapor Phase---(Ref. C-2)

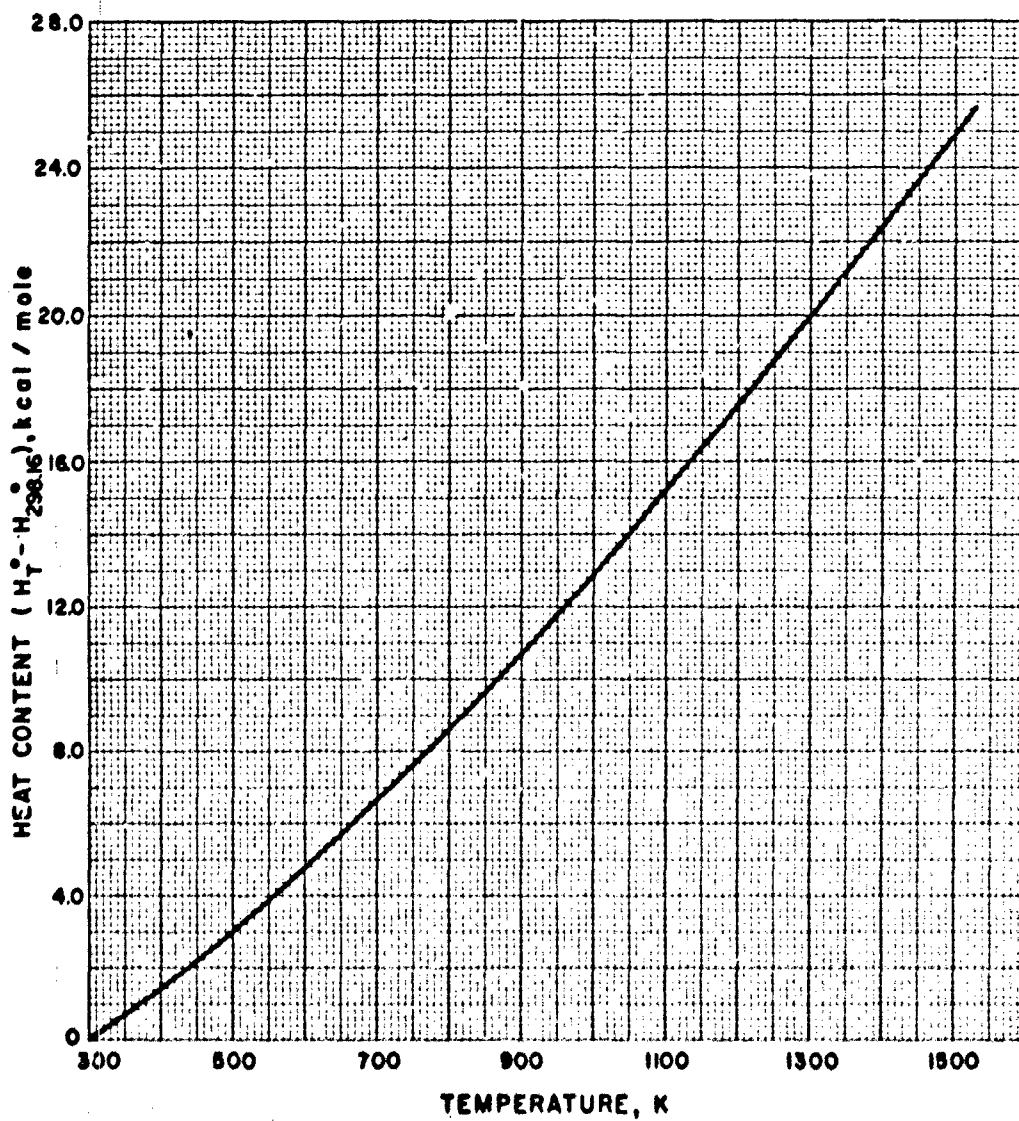


Figure C-6. Enthalpy of Hydrazine--Ideal Gas--(Ref. C-2)

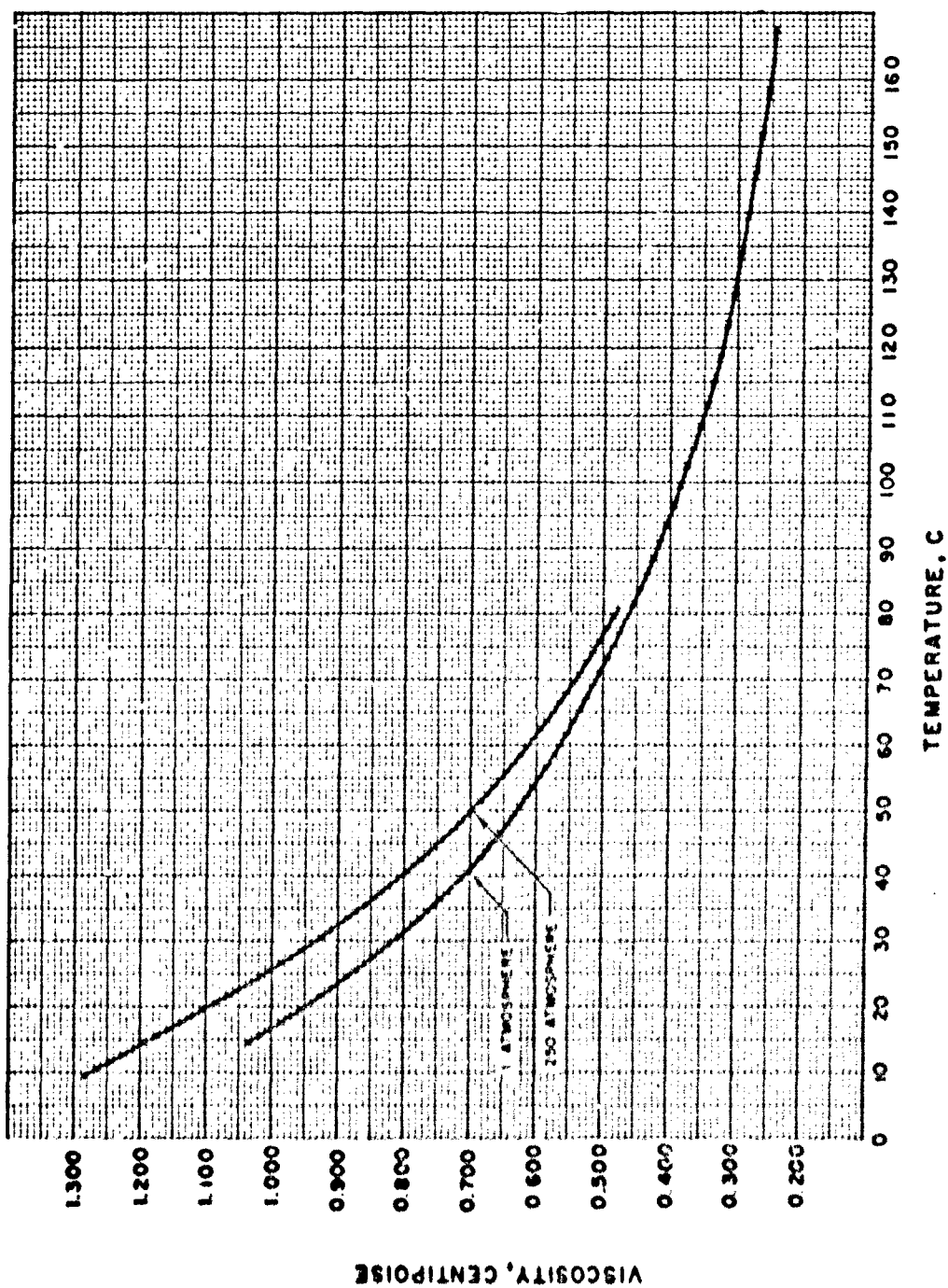


Figure C-7. Viscosity of Anhydrous Hydrazine (Ref. C-9 and C-21)

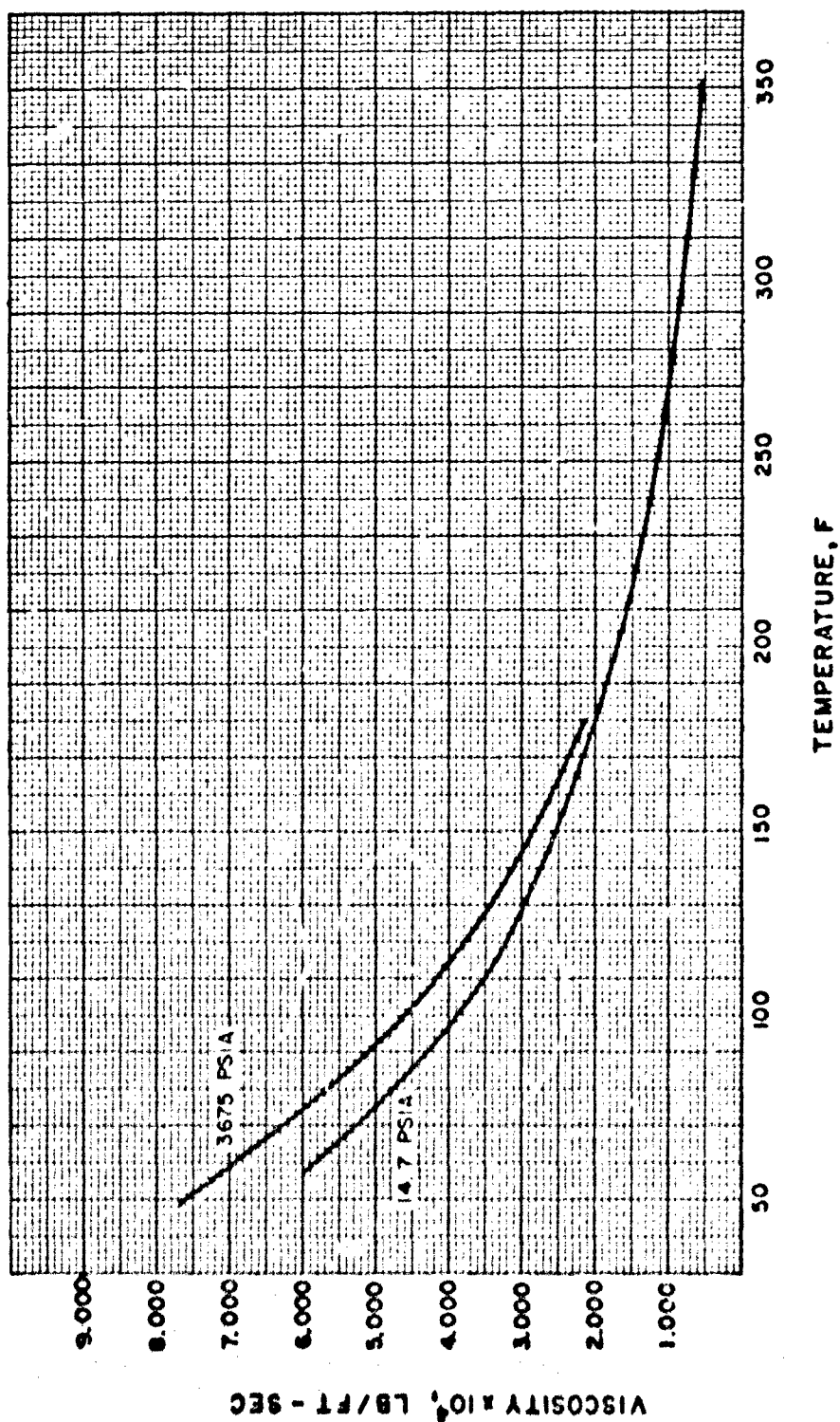


Figure C-7a. Viscosity of Anhydrous Hydrazine (Ref. C-9 and C-21)

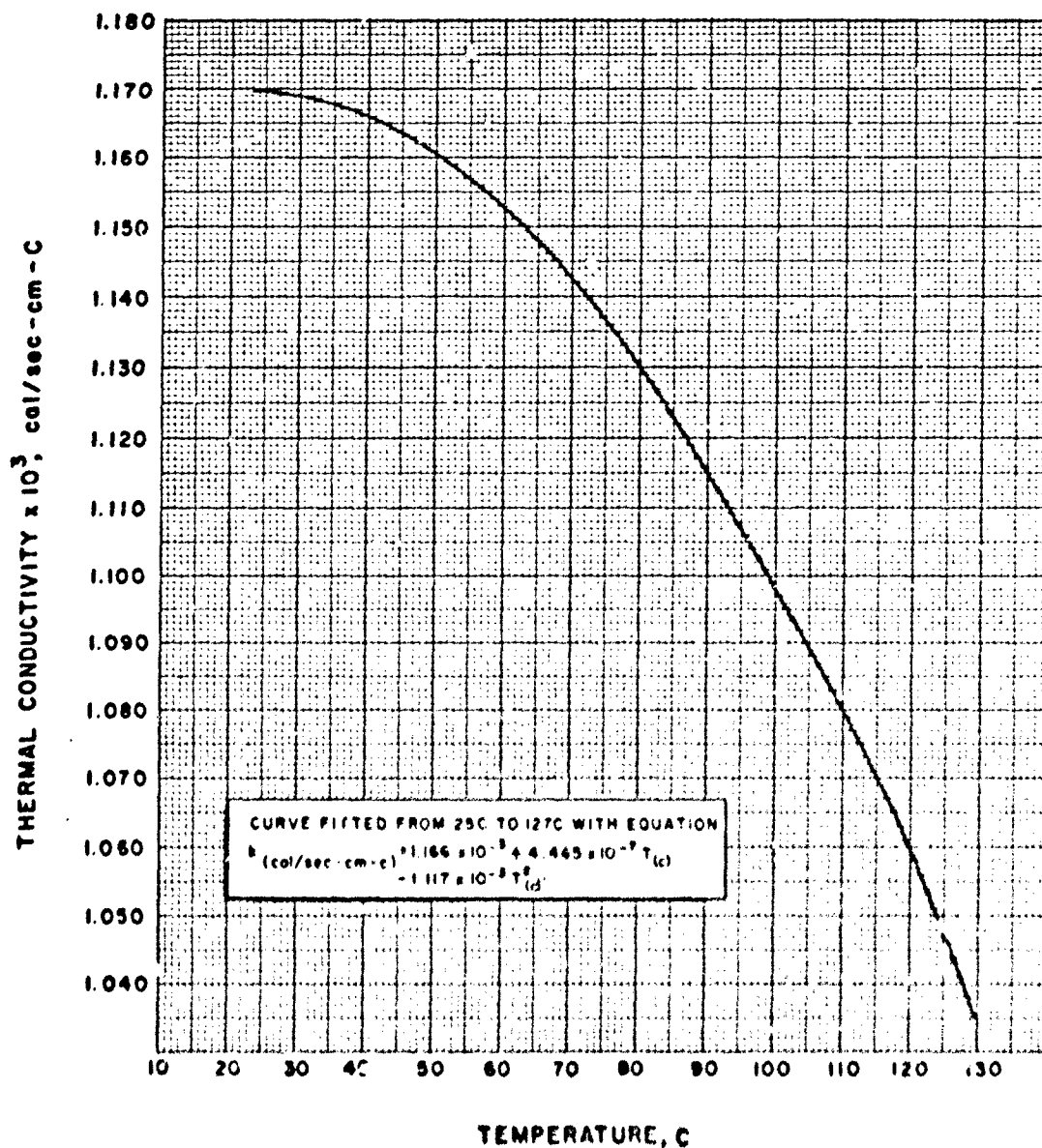


Figure G-8. Thermal Conductivity of Anhydrous Hydrazine
(Ref. G-22)

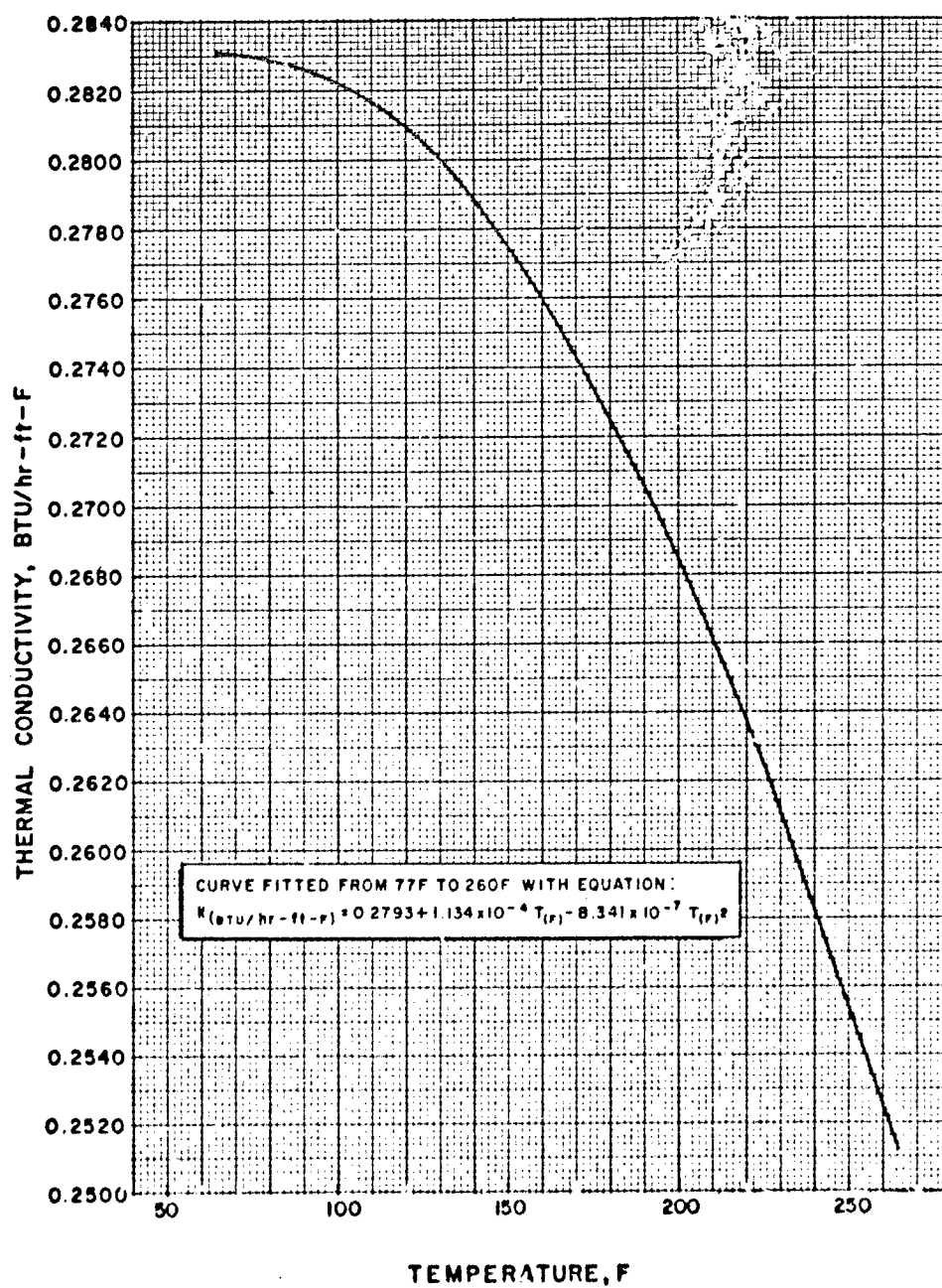


Figure C-8a. Thermal Conductivity of Anhydrous Hydrazine (Ref. C-22)

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3. REPORT TITLE		
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4. DESCRIPTIVE NOTES (Type of report and inclusive dates)		
Interim Final (1 April 1966 through 31 March 1967)		
5. AUTHOR(S) (Last name, first name, initial)		
Constantine, M. T.; Youel, K. J.; Axworthy, A.; Gerhauser, J.; Hamilton, J. V.; Lecce, J. V.; Melvold, R. W.; Quagliano, J.; Rodriguez, S. E.; Seric, M. J.; Unterberg, W.; Williams, M. M		
6. REPORT DATE	7a. TOTAL NO. OF PAGES	7b. NO. OF REFS
1 May 1967	145	13
8a. CONTRACT OR GRANT NO.	9a. ORIGINATOR'S REPORT NUMBER(S)	
AF04(611)-11407	R-7029	
8b. PROJECT NO		
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